University of Alberta

# Investigation of CT beneath MFT Deposition for Oil Sands Tailings Disposal

By

Gengxin Luo

A thesis submitted to the Faculty of Graduate Studies and Research in partial fulfillment of the requirements for the degree of Master of Science

In

Environmental Engineering

Department of Civil and Environmental Engineering

Edmonton, Alberta

Fall 2004

Library and Archives Canada

Published Heritage Branch

395 Wellington Street
Ottawa ON K1A 0N4
Canada

Bibliothèque et Archives Canada

Direction du Patrimoine de l'édition

395, rue Wellington
Ottawa ON K1A 0N4
Canada

*Your file Votre référence*
*ISBN: 0-612-95808-6*
*Our file Notre référence*
*ISBN: 0-612-95808-6*

The author has granted a non-exclusive license allowing the Library and Archives Canada to reproduce, loan, distribute or sell copies of this thesis in microform, paper or electronic formats.

The author retains ownership of the copyright in this thesis. Neither the thesis nor substantial extracts from it may be printed or otherwise reproduced without the author's permission.

L'auteur a accordé une licence non exclusive permettant à la Bibliothèque et Archives Canada de reproduire, prêter, distribuer ou vendre des copies de cette thèse sous la forme de microfiche/film, de reproduction sur papier ou sur format électronique.

L'auteur conserve la propriété du droit d'auteur qui protège cette thèse. Ni la thèse ni des extraits substantiels de celle-ci ne doivent être imprimés ou aturement reproduits sans son autorisation.

---

In compliance with the Canadian Privacy Act some supporting forms may have been removed from this thesis.

While these forms may be included in the document page count, their removal does not represent any loss of content from the thesis.

Conformément à la loi canadienne sur la protection de la vie privée, quelques formulaires secondaires ont été enlevés de cette thèse.

Bien que ces formulaires aient inclus dans la pagination, il n'y aura aucun contenu manquant.

Canada

## Acknowledgements

I would first like to thank Syncrude Canada Ltd., Suncor Energy Inc., and Canadian Natural Resources Ltd., for their generous funding to this project. Without the companies' initiative and financial support, this project would not be a reality.

Despite the many difficulties and problems from the project planning to realisation, as my supervisors, Dr. Selma Guigard and Dr. Phillip Fedorak have shown their relentless support, instruction, and great effort to make this project move forward. Their knowledge and thoughts were among the key factors in making this project run in the right way. The study of this project also benefited very much from Dr. Fedorak's previous experiences of working with oil sands tailings.

Special thanks are to Dr. Mike MacKinnon, from Syncrude Canada Ltd., whose desire to better understand the tailings disposal of CT beneath MFT scheme initiated this project. His expertise, experience, and novel ideas set the general direction for this project.

Without the support of many technical staffs in Environmental Engineering, Biological Sciences, and Geotechnical Engineering groups at the University of Alberta, and Syncrude Research Centre in Edmonton, it would be impossible to make this project run smoothly. Particular thanks are to Debra Long, Garry Solonynko, and Nick Chernuka (Environmental Engineering group) for their routine technical supports in my laboratory work; to Debbi Coy

(Biological Sciences group) for her enthusiasm and commitment in the MPN analysis, and her technical coaching on the MPN analysis for me; to Gerry Cyre and Steve Gamble (Geotechnical Engineering group) for their support in column preparation and frozen column cutting; to Betty Fung (Syncrude Research Centre in Edmonton) for the demonstration in handling the oil sands tailings samples in her laboratory. Also, the supports for sample analyses provided by the Syncrude Analytical Group at the Edmonton Research Centre are much appreciated.

Many students in the Environmental group have given me a timely hand in the need of support, especially the help provided by the students from Dr. Selma Guigard's research group at the time of column filling and assembly.

I appreciate the work completed by Jianmin Pang, who conducted the laboratory work from June 2003, in the Environmental Engineering Laboratory, at the University of Alberta.

Finally, thanks are to all the authors referenced in the thesis.

Gengxin Luo

May 2004

## Table of Contents


*1.0 Introduction* ..... *1*

*2.0 Review of relevant literature* ..... *4*

**2.1 The oil sands industry in Alberta** ..... **4**

**2.2 The bitumen extraction process of oil sands** ..... **5**

**2.3 The fine tailings management** ..... **9**

***2.3.1 The challenges of fine tailings management*** ..... **10**

***2.3.2 The characteristics of MFT*** ..... **12**

***2.3.3 The CT*** ..... **17**

***2.3.4 The options for fine tailings management*** ..... **22**

**2.4 SRB and methanogens** ..... **23**

***2.4.1 The SRB*** ..... **23**

***2.4.2 The methanogens*** ..... **25**

***2.4.3 The competition between SRB and methanogens*** ..... **26**

*3.0 Problem statement and the objective of this project* ..... *28*

**3.1 Problem statement** ..... **28**

**3.2 Objective of this project** ..... **30**

*4.0 Methods and materials* ..... *32*

**4.1 Methodology** ..... **32**

**4.2 Protocol development** ..... **34**

***4.2.1 Number of columns in static column mesocosms*** ..... **34**

***4.2.2 Column design for Systems 1, 2, 3, and 4*** ..... **35**

4.2.2.1 Design of the top covers ..... 35

4.2.2.2 Design of the bottom covers ..... 36

4.2.2.3 Reinforcement of the seals ..... 36

4.2.2.4 Column stopper for Systems 1 and 2 ..... 37

4.2.2.5 Monitoring the interface position of MFT and CT in Systems 1 and 2 ..... 37

***4.2.3 The MFT and CT materials*** ..... **38**

***4.2.4 Procedures for column filling and assembly in Systems 1, 2, 3, and 4*** ..... **38**

***4.2.5 Incubation and regular monitoring of the columns*** ..... **42**

***4.2.6 Sacrificing and freezing procedures of the columns*** ..... **44**

***4.2.7 Frozen column cutting and sampling*** ..... **45**

4.2.7.1 Frozen column cutting and sampling for columns in Systems 1 and 2 ..... 46

4.2.7.2 Frozen column cutting and sampling for columns in Systems 3 and 4 ..... 47

***4.2.8 Subsampling of frozen MFT and CT*** ..... **47**

***4.2.9 Eh measurement on MFT and CT samples*** ..... **48**

***4.2.10 Sulphide analysis in water sample*** ..... **49**

***4.2.11 MPN analysis on MFT and CT samples*** ..... **49**

***4.2.12 Trapped gas analysis on MFT and CT samples*** ..... **50**

***4.2.13 Physical and chemical analyses on water, MFT, and CT samples*** ..... **51**

***4.2.14 Gas analysis by GC*** ..... **52**

4.2.14.1 GC calibration and verification ..... 54

4.2.14.2 Gas analysis by GC for Tedlar bag gas in Systems 1, 3, and 4 ..... 55
4.2.14.3 Gas analysis by GC for in-situ column headspace gas in System 2 ..... 55
4.2.14.4 Gas analysis by GC for trapped gas in MFT and CT ..... 56
*4.2.15 AVS analysis on MFT and CT samples* ..... 56
*4.2.16 Column and sample notations* ..... 58

**4.3 Some definitions and symbols for MFT and CT densifications** ..... 58
*4.3.1 Total volume change (Vt, vol%)* ..... 58
*4.3.2 Total tailings volume change (Vtt, vol%)* ..... 59
*4.3.3 Individual MFT or CT tailings volume change ($V_{MFT}$, $V_{CT}$, vol%)* ..... 60
*4.3.4 Cap water volume (Vcw, vol%)* ..... 61
*4.3.5 Cap water producing rate (Rcw, mL/d)* ..... 61
*4.3.6 Headspace volume (Vhs, mL)* ..... 62

**4.4 Column setup** ..... 62
*4.4.1 Column setup in Systems 1, 3, and 4.* ..... 62
*4.4.2 Column setup in System 2* ..... 65

***5.0 Results*** ..... *68*

**5.1 MFT and CT densifications and water release** ..... 69
*5.1.1 MFT densification and water release in System 3 (Cap water/MFT)* ..... 69
*5.1.2 CT densification and water release in System 4 (Cap water/CT)* ..... 70
*5.1.3 MFT and CT densifications and water release in System 1 (Cap water/MFT/CT)* ..... 72
*5.1.4 MFT and CT densifications and water release in System 2 (Cap water/MFT/CT)* ..... 74

**5.2 Headspace gas analysis** ..... 75
*5.2.1 Tedlar bag gas volume and GC analysis in Systems 1, 3, and 4* ..... 75
*5.2.2 In-situ headspace gas GC analysis in System 2* ..... 75

**5.3 Physical and chemical analyses** ..... 75
*5.3.1 Physical and chemical analyses on baseline MFT and CT* ..... 76
*5.3.2 Physical and chemical analyses in System 3 (Cap water/MFT)* ..... 76
*5.3.3 Physical and chemical analyses in System 4 (Cap water/CT)* ..... 76
*5.3.4 Physical and chemical analyses in System 1 (Cap water/MFT/CT)* ..... 77
*5.3.5 Physical and chemical analyses in System 2 (Cap water/MFT/CT)* ..... 77

**5.4 Trapped gas analysis** ..... 77
*5.4.1 Trapped gas analysis in baseline MFT and CT* ..... 77
*5.4.2 Trapped gas analysis in Systems 1, 2, 3, and 4* ..... 77

**5.5 MPN analysis in System 1** ..... 77

***6.0 Discussion*** ..... *78*

**6.1 MFT and CT densifications and water release** ..... 78
*6.1.1 Total volume change (Vt) in Systems 3, 4, and 1* ..... 78
*6.1.2 Total and individual tailings volume changes (Vtt, $V_{MFT}$, and $V_{CT}$) in Systems 3, 4, 1, and 2* ..... 80
6.1.2.1 Total tailings volume change of MFT (Vtt(3)) in System 3 (Cap water/MFT) ..... 80
6.1.2.2 Total tailings volume change of CT (Vtt(4)) in System 4 (Cap water/CT) ..... 82

6.1.2.3 Total and individual tailings volume changes (Vtt(1), $V_{MFT(1)}$, $V_{CT(1)}$) in System 1 (Cap water/MFT/CT) ..... 83
6.1.2.4 Total and individual tailings volume changes (Vtt(2), $V_{MFT(2)}$, $V_{CT(2)}$) in System 2 (Cap water/MFT/CT) ..... 85
*6.1.3 Cap water volume (Vcw) and cap water producing rate (Rcw)* ..... 90
6.1.3.1 Cap water volume (Vcw) ..... 90
6.1.3.2 Cap water producing rate (Rcw) ..... 90

**6.2 Headspace gas analysis** ..... 91
*6.2.1 Tedlar bag gas analysis in Systems 1, 3, and 4* ..... 91
*6.2.2 In-situ headspace gas analysis in System 2* ..... 100

**6.3 Physical and chemical characteristics of cap water, MFT, and CT** ..... 102
*6.3.1 Physical and chemical characteristics in System 3 (Cap water/MFT)* ..... 103
6.3.1.1 Calcium and magnesium concentrations and the pH ..... 103
6.3.1.2 Sulphate concentration and methanogenesis ..... 104
6.3.1.3 Solids content ..... 106
*6.3.2 Physical and chemical characteristics in System 4 (Cap water/CT)* ..... 106
6.3.2.1 Bromide tracer analysis ..... 106
6.3.2.2 Calcium and magnesium concentrations and the pH ..... 111
6.3.2.3 Sulphate concentration ..... 116
6.3.2.4 Solids content ..... 120
*6.3.3 Physical and chemical characteristics in System 1 (Cap water/MFT/CT)* ..... 121
6.3.3.1 Bromide tracer analysis and the estimation of dilution effect ..... 122
6.3.3.2 Calcium and magnesium concentrations ..... 130
6.3.3.3 Sulphate concentration ..... 136
6.3.3.4 pH and electrical conductivity ..... 142
6.3.3.5 The possible sinks of $SO_4^{2-}$ reduction by SRB and the AVS analysis ..... 143
6.3.3.6 Solids content ..... 146
*6.3.4 Physical and chemical characteristics in System 2 (Cap water/MFT/CT)* ..... 149
6.3.4.1 Bromide tracer analysis ..... 150
6.3.4.2 Calcium and magnesium concentrations ..... 151
6.3.4.3 Sulphate concentration ..... 153
*6.3.5 Comparisons among Systems 1, 3, and 4* ..... 154
6.3.5.1 Comparisons of $Ca^{2+}$, $Mg^{2+}$, and $SO_4^{2-}$ concentrations in the cap water 155
6.3.5.2 Comparisons of pH and electrical conductivity in the cap water ..... 156
6.3.5.3 Comparison of solids content in MFT and CT ..... 156

**6.4 MPN analysis on MFT and CT in System 1** ..... 157
*6.4.1 MPN of methanogens* ..... 157
*6.4.2 MPN of SRB* ..... 158

*7.0 Summary* ..... *160*

**7.1 Total volume change (Vt) in Systems 3, 4, and 1** ..... 160

**7.2 Total tailings volume change (Vtt) in Systems 3, 4, 1, and 2** ..... 160

**7.3 Individual MFT and CT tailings volume change ($V_{MFT}$ and $V_{CT}$) in Systems 1 and 2** ..... 161

7.4 Comparisons of total and individual tailings volume changes (Vtt, $V_{MFT}$, and $V_{CT}$) among Systems 1, 2, 3 and 4 ..... 162
7.5 Cap water volume (Vcw) in Systems 3, 4, and 1 ..... 163
7.6 Cap water producing rate (Rcw) in Systems 3, 4, and 1 ..... 164
7.7 Tedlar bag gas volume in Systems 1, 3, and 4 ..... 164
7.8 GC analysis on the Tedlar bag gas in Systems 1, 3, and 4 ..... 165
7.9 In-situ headspace gas GC analysis in System 2 ..... 166
7.10 Bromide tracer analysis and the dilution effect in System 1 ..... 166
7.11 Calcium and magnesium concentrations in System 1 ..... 167
7.12 Sulphate concentration in System 1 ..... 169
7.13 pH and electrical conductivity in System 1 ..... 170
7.14 Solids content in System 1 ..... 170
7.15 Bromide tracer analysis in System 2 ..... 171
7.16 Calcium and magnesium concentrations in the cap water of System 2 ..... 172
7.17 Sulphate concentration in the cap water of System 2 ..... 173
7.18 Comparisons of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ concentrations in the cap water of Systems 1, 3, and 4 ..... 173
7.19 Comparisons of pH and electrical conductivity in the cap water of Systems 1, 3, and 4 ..... 173
7.20 Comparisons of solids content of MFT and CT among Systems 1, 3, and 4 174
7.21 MPN analysis on the MFT and CT in System 1 ..... 175
*8.0 Conclusion* ..... *176*
*9.0 Recommendations* ..... *178*
9.1 In-situ headspace gas analysis in System 2 ..... 178
9.2 AVS analysis ..... 178
9.3 Trapped gas analysis ..... 178
9.4 The MFT to CT ratio for the CT beneath MFT deposition ..... 179
*10.0 References* ..... *180*
*Appendices* ..... *183*
Appendix A. Methods and materials ..... 184
Appendix B. Results ..... 190
Appendix C. Discussion ..... 234

## List of Tables


*Table 4-1. Static column mesocosm systems.* ..... 32
*Table 4-2. The main parameters for the Micro-GC gas analysis.* ..... 54
*Table 4-3. The planned chemical dosages in CT for Systems 1 and 4.* ..... 63
*Table 4-4. MFT and CT mixings and baseline sample notations for Systems 1, 3, and 4.* ..... 64
*Table 4-5. The planned chemical dosages in CT of System 2.* ..... 66
*Table 4-6. MFT and CT mixings and baseline sample notations for System 2.* ..... 67
*Table 5-1. Incubation time and sacrificed columns for Systems 1, 2, 3, and 4.* ..... 68
*Table 5-2. Summary of relevant parameters at the end of specific incubation period in System 3.* ..... 69
*Table 5-3. Summary of relevant parameters at the end of specific incubation period in System 4.* ..... 71
*Table 5-4. Summary of relevant parameters at the end of specific incubation period in System 1.* ..... 73
*Table 5-5. Summary of relevant parameters at the end of 1 y of incubation in System 2.* ..... 75
*Table 6-1. Tailings volume change and solids content of MFT in System 3 at specific incubation times.* ..... 81
*Table 6-2. Tailings volume change and solids content of CT in System 4 at specific incubation times.* ..... 82
*Table 6-3. Total and individual tailings volume changes and solids content at specific incubation times in System 1.* ..... 84
*Table 6-4. Total and individual tailings volume changes at specific incubation times in System 2.* ..... 88
*Table 6-5. The $CH_4$ yield in Systems 1, 3, and 4.* ..... 94
*Table 6-6. The $Br^-$ mass balance calculation in System 4.* ..... 110
*Table 6-7. The $Ca^{2+}$ mass balance calculation in System 4.* ..... 113
*Table 6-8. The ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in the cap water and porewaters of System 4 at column sacrificing.* ..... 115
*Table 6-9. The $SO_4^{2-}$ mass balance calculation in System 4.* ..... 118
*Table 6-10. The $Br^-$ mass balance calculation in System 1.* ..... 125
*Table 6-11. The volume percent (vol%) of CT water with balance MFT water, based on $Br^-$ analysis in System 1.* ..... 127
*Table 6-12. The estimation of dilution effect ($P_d$) at reducing $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ concentrations, and the EC for the cap water of System 1.* ..... 129
*Table 6-13. The $Ca^{2+}$ mass balance calculation in System 1.* ..... 133
*Table 6-14. The ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in the cap water and porewaters of System 1 at column sacrificing.* ..... 135
*Table 6-15. The $SO_4^{2-}$ mass balance calculation in System 1.* ..... 139
*Table 6-16. AVS analysis and related $SO_4^{2-}$ and $S^{2-}$ measurements in System 1.* ..... 145

## List of Figures

*Figure 2-1. Schematic of oil sands processes at Syncrude and Suncor.* ..... 8
*Figure 4-1. Schematics of side view of the column stopper used for filling columns in Systems 1 and 2* ..... 39
*Figure 4-2. Schematics of column filling procedures for Systems 1 and 2* ..... 42
*Figure 4-3. Scheme of frozen column cuttings with the shaded areas to be cut off* ..... 46
*Figure 4-4. The columns of Systems 1, 3, and 4 in the incubation room.* ..... 65
*Figure 4-5. The columns of System 2 in the incubation room.* ..... 67
*Figure 5-1. Relative heights of cap water and MFT in System 3.* ..... 70
*Figure 5-2. Relative heights of cap water and CT in System 4.* ..... 72
*Figure 5-3. Relative heights of cap water, MFT, and CT in System 1.* ..... 74

## List of Abbreviations

| | |
|---|---|
| AVS | acid volatile sulphide |
| BDL | below detection limit |
| CT | composite tailings or consolidated tailings |
| CT-L | lower zone of the CT layer |
| CT-U | upper zone of the CT layer |
| d | day |
| EC | electrical conductivity |
| Eh | redox potential |
| g | gram or gravity |
| GC | gas chromatography |
| H | height |
| He | Helium |
| h | hour |
| ID | inside diameter |
| L | litre |
| M | mole per litre |
| MFT | mature fine tailings |
| MFT-L | lower zone of the MFT layer |
| MFT-U | upper zone of the MFT layer |
| mg | milligram |
| mL | millilitre |
| MPN | most probable number |
| ms | millisecond |
| NA | not available |
| nd | non-detectable |
| nL | nanolitre |
| NST | non-segregating tailings |
| OD | outside diameter |
| ORP | oxidation - reduction potential |
| s | second |
| SRB | sulphate-reducing bacteria |
| $t_{incu}$ | incubation time |
| $t_R$ | retention time |
| USEPA | United States Environmental Protection Agency |
| wk | week |
| y | year |

## 1.0 Introduction

The two original commercial oil sands companies, Syncrude Canada Ltd. and Suncor Energy Inc., have used a caustic-based Clark Hot Water Extraction process to recover bitumen from oil sands in Alberta, Canada, since the late 1960's. This process produces large quantities of aqueous slurry, called extraction tailings. The extraction tailings are a mixture of solids, water, salts, and unrecovered bitumen (MacKinnon 1989). The tailings are not released to the environment, but are stored in large settling basins (also called tailings ponds). Once in the tailings ponds, the fast-settling coarse sands fraction settles out on the beaches and is used to construct dikes for the tailings ponds. The remaining muddy liquids make up the fine tailings, which consist of slow-settling clay and silt particles, dissolved inorganic and organic substances, residual bitumen, and water.

The fine tailings are allowed to dewater and densify over time in the tailings ponds. After 2 to 3 y of relatively rapid densification a dense mature fine tailings (MFT) zone will be produced at the bottom of the tailings ponds. The released water on the top layer of the tailings ponds is reused for extraction process (List and Lord 1997). The MFT has a solids content of greater than 30 wt%. The dewatering and densification of MFT under natural conditions are very slow, with consolidation into a trafficable consistency expected to take hundreds of years (FTFC 1995).

The large volume of MFT poses a big challenge to oil sands companies. To increase the MFT densification rate and reduce the inventory of existing

MFT, the oil sands companies (Syncrude and Suncor) have proposed the CT (Composite Tailings in Syncrude and Consolidated Tailings in Suncor) technology (FTFC 1995). In CT production, the MFT, coarse solids, and gypsum are mixed together. The addition of gypsum causes the clay particles to aggregate and the slurry viscosity to increase. Thus, the fine solids and coarse solids would stay together at deposition, creating non-segregating tailings (NST) (Matthews *et al.* 2000). The initial solids content of CT is approximately 60 wt%. When the CT slurry is deposited, the CT deposit starts dewatering immediately. The CT deposit will densify to a solids content of approximately 75 wt% within a few weeks or months. CT will become trafficable within a few years, leading to a dry landscape at site restoration. The CT release water has to be recycled back to the plant, primarily for the extraction process. It is essential that this CT water not to be detrimental to bitumen recovery efficiency and plant integrity. The CT technique has been under research since 1990 (List and Lord 1997).

In the CT process, the optimum dosage of gypsum would be around 1000 mg/L (i.e. 1 kg gypsum per $m^3$ of CT slurry) (Matthews *et al.* 2000). As a result of the high gypsum dosage, the CT release water contains a high concentration of calcium ($Ca^{2+}$) and sulphate ($SO_4^{2-}$) (MacKinnon *et al.* 2000). These high concentrations of $Ca^{2+}$ and $SO_4^{2-}$ could decrease bitumen recovery and cause other process problems when reused for process recycling. To address this issue, the oil sands companies have been examining various treatment options, among which is the co-disposal of CT and MFT, with the CT being

placed beneath MFT. It is expected that this CT beneath MFT deposition will take advantages of physical, chemical, and anaerobic microbiological processes occurring in the CT and MFT layers to remove or reduce the high concentrations of $Ca^{2+}$ and $SO_4^{2-}$ in the CT release water. At Syncrude Canada Ltd., a pilot-scale field demonstration test was conducted in 1995 to study this CT beneath MFT deposition. The purposes of this CT beneath MFT deposition study were to improve the CT release water quality and MFT densification rate, and to see if the CT densification rate will be adversely affected (Shaw *et al.* 2001). The field demonstration test indicated that the CT beneath MFT deposition initiated changes that were beneficial to tailings disposal, with respect to the release water quality and densification rates of both MFT and CT (Shaw *et al.* 2001). However, prior to recommending this CT beneath MFT deposition scheme as a viable full-scale tailings disposal and management option, it is necessary to conduct a controlled laboratory experiment, to understand the physical, chemical, and microbiological processes that occur in this co-disposal tailings option as the CT release water goes through the MFT layer (Shaw *et al.* 2001).

The purpose of this project is thus to demonstrate whether the placement of CT beneath MFT is a feasible and beneficial disposal technology in tailings management under the laboratory conditions, with respect to the cap water quality and densification rates of both the MFT and CT.

## 2.0 Review of relevant literature

### 2.1 The oil sands industry in Alberta

Oil sands are geological units that consist of a mixture of bitumen, formation water, coarse sands, and fine particles (clays and silts). The oil sands deposits in Alberta contain an estimate of over 1.7 trillion barrels of bitumen, of which approximately 300 billion barrels are recoverable with existing technologies. These currently recoverable reserves are similar in size to the proven reserve of Saudi Arabia (ACR 1995).

There are four major reserves of oil sands in the province of Alberta: Athabasca, Peace River, Wabasca, and Cold Lake reserves. The Athabasca oil sands deposit is located in northeastern Alberta, and by itself is the largest petroleum resource in the world. Crude oil from oil sands currently accounts for over 30% of Canada's domestic oil production, and is expected to grow to more than 50% by year 2005 (Syncrude 2000a). Canada's energy supply can be secured for more than 200 years if Alberta's oil sands are fully developed (Syncrude 2000b).

Serious exploration of oil sands began in the early 1920s by the Alberta Government. Various entrepreneurs have explored the commercial potential of the vast oil sands. In 1967, Suncor Energy Inc. opened the first commercial-scale surface oil sands processing facility for mining the oil sands and extracting and upgrading the bitumen at its Lease 86 at the Tar Island site on the Athabasca River, about 30 km north of Fort McMurray in northern Alberta (MacKinnon and Sethi 1993). In the same area, Syncrude Canada Ltd. started production in

1978 at its Leases 17 and 22, located about 1.5 km west of the Athabasca River. Syncrude Canada Ltd. is now the world's largest producer of crude oil (known as Syncrude Sweet Blend) from oil sands. Syncrude Canada Ltd. and Suncor Energy Inc. are currently mining the oil sands using the surface mining operation, and extracting and upgrading the bitumen at the Athabasca oil sands deposit. The two companies produce over $500 \times 10^3$ barrels of crude oil daily from about $1.0 \times 10^6$ tons of ore processed (Dr. MacKinnon, Syncrude Canada Ltd., personal communication).

**2.2 The bitumen extraction process of oil sands**

Oil sands can vary in grade, fines or clay content, mineralogy, and salt content. The typical oil sands contain an average of approximately 10 wt% of bitumen; the remainder contains coarse sands (>22μm in diameter), fine particles (<22μm in diameter), and water (FTFC 1995).

The Athabasca oil sands are water-wet. The bitumen is not in direct contact with the sands or clay surface, but is separated by a thin layer of water. Hot water extraction has been proven to be a suitable method for removing and recovering the bitumen from the oil sands.

Bitumen is extracted from the oil sands in the extraction plant. The extraction process used by Syncrude and Suncor is based on the Clark hot water extraction process, which was developed by Dr. Karl Clark in the 1920s.

In the Clark hot water extraction process, the oil sands are slurried and conditioned for bitumen separation in large horizontally rotating tumblers by

mixing with hot water, steam, and caustic soda (NaOH). The introduction of steam is to raise the temperature of the slurry in the mixing tumbler to about 80°C. The addition of the caustic soda will raise the pH of the mixture to the range of 9.0 to 11.0. The slurry usually stays in the tumblers for less than 10 min. At this conditioning stage, bitumen is dislodged from the sand particles, resulting in a loose association of bitumen, sand, and water.

The slurry from the tumblers is then discharged onto vibrating screens to remove large size particles, such as rocks and lumps of undigested oil sands and clay. The slurry produced at this stage is further diluted with additional quantities of hot water and pumped to the primary separation vessels.

In the primary separation vessels, the bitumen floats to the surface as primary froth, while the coarse sands settle out to the bottom as primary tailings. The middlings (from the central portion in the primary separation vessels) and the primary tailings are combined and pumped to the tailings oil recovery vessels (the secondary separation vessels) to further recover most of the remaining bitumen, as the secondary froth.

The secondary froth is combined with the primary froth. This froth mixture is first deaerated and heated, then is fed to the froth treatment plants. Both Syncrude and Suncor's bitumen froth products contain large quantities of water and fine solids (mainly clays), which must be removed before proceeding to the upgrading process. Froth treatment minimises the water and solids going to the upgraders.

During the upgrading process, bitumen is converted from a viscous, tar-like material to a low-sulphur synthetic crude oil (a light gold-coloured liquid), using a coking and hydrocracking process. The resulting synthetic crude oil is then transported by pipeline to the conventional refineries for further refining.

Figure 2-1 shows the schematic flow diagram of the oil sands processes from open surface mining to synthetic crude oil production (FTFC 1995).

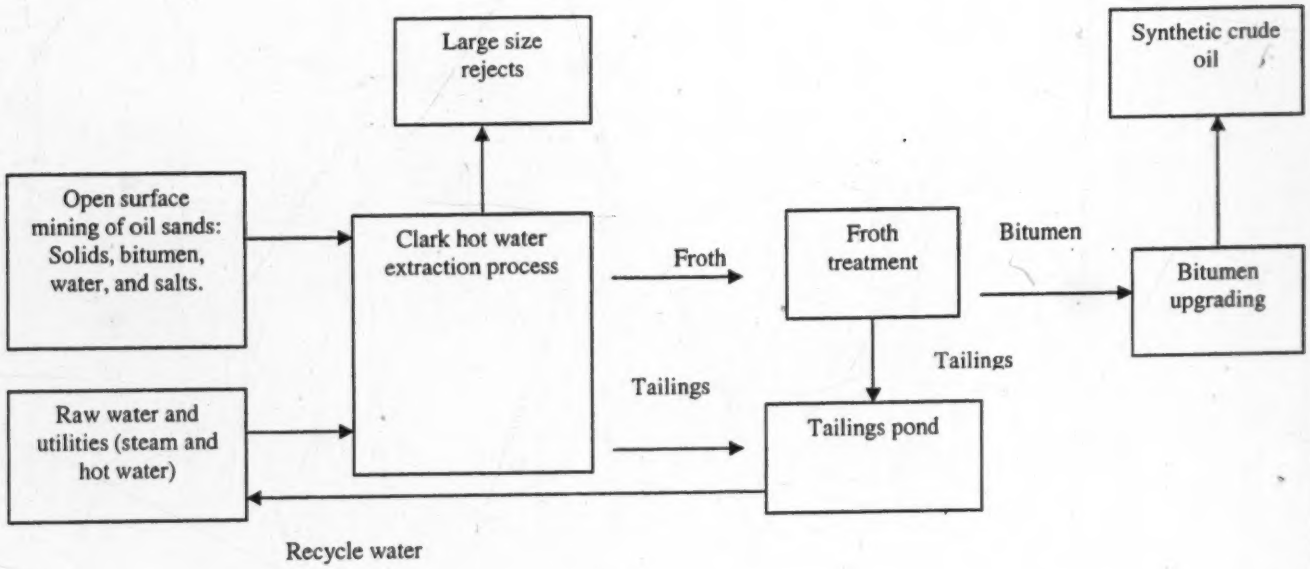



**Figure 2-1. Schematic of oil sands processes at Syncrude and Suncor** (adapted from FTFC 1995).

## 2.3 The fine tailings management

One of the main disadvantages of the aqueous digestion processes is its by-product of a large quantity of slurry tailings, which contain water, a coarse sands fraction, a fines fraction, and unrecovered bitumen (FTFC 1995). Neither the tailings solids nor the released water can be discharged into the environment, so that the present development is operating under a "zero discharge" policy in tailings management.

All the tailings from the primary and secondary separation vessels and the froth treatment plant are discharged into large settling basin (i.e. tailings ponds), as the "total extraction tailings" with solids content between 40 and 55 wt% (Dr. MacKinnon, Syncrude Canada Ltd., personal communication). Upon deposition, most (>95%) of the coarse solids (the sands fraction with particle diameter >22 μm, so is also called tailings sands) segregate and settle out from the fines fraction (<22 μm in particle diameter) and are used to build dykes that surround the tailings ponds. A large fraction (about 50%) of the fines are entrained with the coarse solids and settle out with them. The remainder of fines (fine tailings), consisting mainly of slow-settling fine silt and clay particles, water, and residual bitumen, are carried into the tailings ponds as thin fine tailings, with a solids content of about 3 to 8 wt% (MacKinnon and Sethi 1993). The "total extraction tailings" from the oil sands extraction process are thus segregated into tailings sands and fine tailings, which are disposed of separately.

In the tailings ponds, the thin fine tailings settle rapidly to about 20 wt% solids content over a period of several months, and then densify slowly over 2 to

3 years to approximately 30 wt% solids content (Mikula *et al.* 1996). The fine tailings with solids content larger than 30 wt% are termed "mature fine tailings", i.e. MFT (FTFC 1995). The produced surface water (i.e. the released water) in the tailings ponds is low in suspended solids (from 0.1 to 1 wt% at Syncrude and Suncor) and is reused as the recycle water in the extraction plant (MacKinnon and Sethi 1993).

### *2.3.1 The challenges of fine tailings management*

In tailings management, technologies for handling coarse solids (the tailings sands) exist. The biggest difficulty in tailings management is the disposal of fine tailings.

In the fine tailings disposal and management, there are two major challenges. One is the slow self-weight densification rate of the fine tailings. Unlike the tailings sands, which segregate and settle out quickly from the tailings slurry, the fine tailings densify at a slow rate due to the nature of the fine particles (mainly kaolinite and illite clays) and the chemistry of the porewater. The high $Na^+$ content of the fine tailings porewater will cause clay particles to repel each other and remain in suspension in the tailings water, thus preventing the fine tailings from settling (Dr. MacKinnon, Syncrude Canada Ltd., personal communication). After 2 to 3 y of relatively rapid initial settling and densification, the fine tailings will reach a solids content of approximately 30 wt% and become MFT. Further densification of the MFT is very slow. It is

estimated that the natural densification of MFT to a trafficable surface would take hundreds of years (FTFC 1995).

Based on Syncrude's tailings pond data, an empirical equation for describing the solids content increase of fine tailings over time under self-weight conditions is given below (Mikula *et al.* 1996):

$$S_c = 18.6 + 6.35 \ln y \quad \text{.....(2-1)}$$

where $S_c$ is the solids (<22 μm in particle diameter) content (wt%); y is the number of years of settling.

The production and accumulation of large volumes of fine tailings is another challenge. The Clark hot water extraction process uses large volumes of water and thus results in the production of large quantities of fluid wastes, i.e. the fine tailings. Because of the high water content in the fine tailings, the fine tailings retain fluid characteristics and must be stored behind geotechnically secure dikes with little possibility of being used as a solid substrate for plant establishment (FTFC 1995). The accumulation and disposal of the fine tailings have been on going since 1967 at Suncor and since 1978 at Syncrude. It is not allowed to discharge the fine tailings and its released water to the surrounding environment. All the fine tailings are stored in on-site large tailings ponds. Consequently, a large inventory (more than 500 million $m^3$) of fine tailings has been accumulated in the tailings ponds at Syncrude and Suncor, in Fort

McMurray, Alberta (Xiaomei and Yongsheng 1995). The Mildred Lake Settling Basin is Syncrude's largest settling basin and covers about 25 $km^2$, with a water surface area of about 12 $km^2$. Suncor's four tailings ponds cover a total area of about 13 $km^2$, with a water surface area of about 7 $km^2$ (FTFC 1996). Fine tailings are being accumulated at a rate of approximately 20 million $m^3/y$ (Xiaomei and Yongsheng 1995).

Storage and disposal of the large volumes of fine tailings are considered to be the major environmental challenge facing the surface mining oil sands industry (AERCB 1984). For this reason, considerable efforts have been made by the oil sands companies, government, and research institutions to identify suitable technologies for reclaiming the existing fine tailings inventories and for reducing the future volumes of fine tailings that are being continuously produced (FTFC 1995).

### *2.3.2 The characteristics of MFT*

MFT are mainly comprised of fine mineral solids, water, and residual bitumen. The solids content of MFT is larger than 30 wt%. The majority of fine mineral solids are clays (<2 μm in particle diameter), the remainder being silts (from 2 to 22 μm in particle diameter) and fine sands (FTFC 1995). Residual bitumen content (defined as weight of bitumen divided by weight of minerals) is typically in the range of 2 to 10 wt% (FTFC 1995).

There are two models to explain the slow densification rate of MFT: the bitumen model and the mineral model. In the bitumen model, the slow densification of MFT slurry is attributed to the presence of residual bitumen and soluble organic surfactants. They bind the clay particles into a stable aggregate structure that is slow to dewater and consolidate. In the mineral model, the water holding capacity and stability of MFT are attributed to the presence and interaction of fine mineral clays. The clay particles interact with each other and form a three-dimensional gel-like network structure (a "card house-like" structure) in the presence of electrolytes. Other MFT components (bitumen, water, coarser solids) are trapped in the network. Investigations using cryogenic electron microscopy show a definite structure of the clay mineral flocs or aggregates (Mikula *et al.* 1996). The densification rate of MFT is controlled by the permeability and compressibility of its structure. Studies since 1991 have favoured the mineral model in accounting for the MFT stability (Mikula *et al.* 1996).

According to the mineral model of MFT structure and stability, the bulk volume and water holding capacity of MFT are determined by the mineral components (FTFC 1995). The amount of ultra-fines in the particle diameter range of 0.2 to 0.3 μm (which comes from the original oil sands ores) in MFT will account for more than 90% of its water holding capacity (FTFC 1995). Furthermore, the bulk properties of MFT are a function of the chemistry of the porewater (FTFC 1995). The MFT from Clark hot water extraction process is a

weakly flocculated suspension of sodium-rich clays that densifies slowly by self-weight settling.

Tang (1997) reviewed and examined the MFT microstructures using scanning electron microscope (SEM). The MFT has a highly dispersed three-dimensional card-house structure (also called gel structure) with clay particles aligned in edge-to-edge and edge-to-face patterns. The porewater chemistry of MFT, including pH, alkalinity, salinity, ionic content, and organic matter, may contribute to the formation and stability of the MFT structure. The residual bitumen in MFT could also reduce the MFT hydraulic conductivity, and hence reduce the MFT densification rate. The high water holding capacity and slow densification rate of MFT may be explained by this gel structure.

Chemical, physical, and toxicological properties of the fine tailings and its porewater are a function of the source and composition of the oil sands ores. In addition, both the make-up water used for plant process and the chemicals added as process aids during hot water extraction can add extra inorganic and organic compounds to the fine tailings stream (FTFC 1995). The composition of the water in the tailings ponds is not static. As all the process water and wastes are stored in the tailings ponds and the released water is recycled, the ions in the water of tailings ponds accumulate. Over time, the recycle water from the tailings ponds becomes more brackish (Mikula *et al.* 1996).

The major anions ($Cl^-$, $SO_4^{2-}$, $HCO_3^-$) and cations ($Na^+$, $K^+$, $Mg^{2+}$, $Ca^{2+}$) account for most of the dissolved fraction in the MFT porewater. Sodium is the predominant cation, accounting for approximately 95% of the cation equivalents

(defined as ion concentration divided by ion equivalent weight) in the MFT water fraction. Most of the sodium comes from the process chemicals. The other cations ($K^+$, $Mg^{2+}$, $Ca^{2+}$) are minor components that represent only about 5% of the total cation equivalents (MacKinnon 1989).

Bicarbonate is the most abundant anion and accounts for 50 to 75% of the anion equivalents. The pH of the MFT porewater ranges from 7.3 to 8.3 (Dr. MacKinnon, Syncrude Canada Ltd., personal communication). In this pH range, the dissolved inorganic carbon is present predominantly as $HCO_3^-$, which determines the pH in the MFT slurry. Sulphate concentration is affected by microbial processes in anaerobic environment, where the $SO_4^{2-}$ is reduced to $S^{2-}$ by sulphate-reducing bacteria (SRB). The $Na^+$ and $Cl^-$ ions are conservative species and are little affected by microbiological or chemical interactions (MacKinnon 1989).

The major hydrocarbon components of fine tailings include unrecovered bitumen and the naphtha. The concentrations of water-soluble organics released during the hot water extraction process in both the surface water and pore water of the tailings ponds are fairly low (55 to 85 mg/L) (FTFC 1995). Fine tailings and the associated water are toxic to aquatic biota. Acid-extractable organics account for most of the acute toxicity in tailings pond recycle water. Up to 95% of the total acid fraction extractable from fine tailings is composed of naphthenic acids (FTFC 1995).

The concentrations of oxygenated nutrients (such as nitrite, nitrate, and orthophosphate) in the MFT porewater and produced cap water are low. Most of

the nitrogen is present as ammonia in the range of 2 to 6 mg/L (MacKinnon 1989).

The concentrations of dissolved trace metals in fine tailings water are low relative to regulatory guidelines for the protection of freshwater aquatic biota and have been maintained quite constant over time. Usually only aluminum (Al), barium (Ba), boron (B), iron (Fe), molybdenum (Mo), strontium (Sr), and zinc (Zn) have concentrations above 0.1 mg/L. The trace metal level in the fine tailings does not appear high enough to be detrimental to the environmental acceptability of water quality (MacKinnon 1989).

The anaerobic tailings ponds are rich in microorganisms. Methanogens are active in the Mildred Lake Settling Basin (operated by Syncrude Canada Ltd.) (Holowenko 2000). The production of methane may have detrimental effects on the reclamation of the tailings ponds by affecting the tailings settling behaviour, producing fugitive emission of low molecular hydrocarbons, and leading to anaerobic conditions in the cap water (Fedorak *et al.* 2000). SRB are an important group of microorganisms in anaerobic ecosystems that have sulphate as the terminal electron acceptor. Methanogens and SRB are two important microorganisms affecting the reclamation of the tailings ponds and MFT slurry (Fedorak *et al.* 2000).

The typical strength values of vane shear test of MFT are in the range of 0.2 to 2.5 kPa (FTFC 1995). These values are very low relative to the typical geotechnical range for consolidated solids. Therefore, MFT is too weak to

support a trafficable surface (FTFC 1995). Dewatering of MFT occurs slowly, taking hundreds of years to develop into a structure of solid soil (FTFC 1995).

### *2.3.3 The CT*

The majority of the fine minerals (mainly clays) in the oil sands tailings stream segregates from the coarse sands fraction and form the slurry fine tailings in the tailings ponds. After 2 to 3 y of relatively rapid settling, the fine tailings become a slow settling MFT. Because of the large volumes and the fluid character that requires geotechnically stable enclosure, the fine tailings and MFT pose the biggest environmental challenge to the oil sands companies in reclaiming the disturbed areas and tailings ponds.

It is thus a desirable option for the oil sands industry to prevent the segregation of coarse sands and fine minerals as the extraction tailings are deposited into the tailings ponds, such that the fines are incorporated in the sands deposit as they were in the original ore body (FTFC 1995). To prevent fines segregation, it is necessary to modify the tailings stream by increasing the coarse sands content, increasing the fines content, and adding a chemical coagulant (FTFC 1995). The floc structure of untreated MFT from Clark hot water extraction process is not strong enough to support a load or stress that would allow rapid densification by gravity. A chemical treatment can change the weak floc structure of the MFT into a stronger floc structure that would support some surcharge and enhance the densification (FTFC 1995).

Non-segregating tailings (NST) are a tailings mixture in which the fines and coarse sand particles settle simultaneously to form a uniform deposit. The introduction of coarse solids imparts an internal stress that significantly enhances the densification rate of the NST. The mixture of coarse sands tailings and fine tailings requires the presence of chemical additives (coagulants) to produce a non-segregating behaviour. With the addition of polyvalent cations (e.g. $Ca^{2+}$ from gypsum), the fine particles (clays) in oil sands tailings will coagulate, resulting in the possibility to produce a MFT floc structure that is strong enough to support a surcharge and hence to create a non-segregating tailing (NST). Calcium ion has been known to be an effective coagulant in modifying the MFT properties. The addition of calcium can be achieved by the addition of gypsum ($CaSO_4 \bullet 2H_2O$) (FTFC 1995).

The term of CT has gained its wide use in replace of NST since 1995 (Shaw *et al.* 1996). Large-scale field testings of CT approach were started in 1993/1994 at Suncor and in 1995 at Syncrude (List and Lord 1997). In 1996, Suncor began to use CT technology based on gypsum treatment on a commercial scale to reduce the large volume of fine tailings in its tailings ponds. Suncor plans to use the CT process as the basis for future tailings management. In 1997/1998, Syncrude successfully demonstrated a full-scale prototype operation of CT test, also utilising gypsum as the coagulant. Under the present plans, a large portion of the extraction tailings will be handled using the CT process in Syncrude (MacKinnon *et al.* 2000).

In the CT process at Syncrude Canada Ltd., the densified coarse tailings sands (obtained by hydrocycloning the extraction tailings) are combined with MFT and a chemical coagulant aid to form a type of NST. Unlike the conventional extraction tailings, when the CT is deposited, the fines will stay and settle together with the coarse sands fraction. The coarse sands act as a stress or load that enhances the settling of the deposit. CT deposit has a higher permeability than MFT, resulting in more rapid dewatering (MacKinnon *et al.* 2000).

Factors affecting the segregation of CT mixture include total solids content (density), fines content (a fraction of the total solids), particle size gradation, and type and dosage of coagulant aids. These factors can be manipulated individually or in combination to shift the segregation boundaries (Matthews *et al.* 2000).

Three means of slurry manipulation have been evaluated in the course of the CT development; these are increase in solids content by hydrocyclone densification of the extraction tailings, increase in fines content through enrichment with MFT, and chemical adjustment by addition of coagulants (Matthews *et al.* 2000).

Without the addition of chemical coagulants, the tailings would exhibit a gap-graded particle size distribution and lead to the segregation of the fines from the coarse solids during the discharge and deposition. The addition of coagulant aids to the CT mixture (consisting of coarse tailings and MFT) is an essential component of the CT process. The coagulants produce changes in the properties

of clays in the CT mixture and cause the colloidal particles to aggregate. This phenomenon has the effect of changing the particle size distribution of the solids in the CT mixture to provide a more uniformly graded mixture that can suppress or eliminate segregation (Matthews *et al.* 2000).

During the development of the CT process, various coagulant aids were assessed, including sulphuric acid, lime (CaO, $Ca(OH)_2$), acid-lime ($H_2SO_4$-CaO), gypsum, sodium aluminate ($Na_2Al_2O_3$), alum (48% $Al_2(SO_4)_3 14.3H_2O$), and organic polymers (polyacrylamides). The segregation characteristics, deposit dewatering and densification rates, released water quality, and economics are the factors in the assessment and selection of coagulants. Based on on-going research efforts, gypsum has proven to be a robust, effective, easy to handle, and readily available coagulant aid to the CT process. As a result, Syncrude used gypsum as the coagulant for the 1995 field demonstration and the 1997/1998 prototype CT test. Through these evaluations, Syncrude, as well as Suncor, have chosen gypsum as the base coagulant for the future commercial application of CT. The optimum gypsum dosages are in the range of 1000 to 1200 $g/m^3$ of tailings (Matthews *et al.* 2000).

In the CT mixture, most of the fines and porewater come from the MFT component. This is an important factor when predicting the quality of CT release water. The optimum solids content of CT is about 60 wt% (Dr. MacKinnon, Syncrude Canada Ltd., personal communication). When the CT slurry is deposited and starts settling, the CT deposits experience two stages of densification: the first stage of initial settling and the second stage of long-term

self-weight consolidation. The initial settling starts immediately upon deposition of the CT mixture. During this initial settling period, the CT deposit undergoes a substantial volume reduction as its porewater being released. As the CT deposit densifies to a solids content of 75 wt% in weeks or months, approximately 50% of the initial water can be released. The rate of the initial settling depends on the amount and type of fines, chemical addition, mixing procedure, and the hydraulic conductivity of MFT. The long term consolidation of CT begins as the sand grains become in contact with one another and form a sand matrix. Excess water is slowly released from the CT as it consolidates over a long-term (MacKinnon *et al.* 2000; Tang 1997). If allowered to dewater and drain, the CT deposit will become trafficable in a few years.

In the CT process, the gypsum dosage at about 1000 g per $m^3$ of CT mixture would result in approximately 1000 mg/L of sulphate ($SO_4^{2-}$) in the CT porewater (MacKinnon *et al.* 2000). With the high $SO_4^{2-}$ concentration in the CT porewater, the CT will inhibit methane production by creating an environment that is more suitable for SRB and so that the SRB would out-compete the methanogens. Fedorak *et al.* (2002) has shown that the addition of sulphate to fine tailings decreased methane production, and the methanogenesis appeared to have started to a large extent only after the sulphate concentration has dropped to approximately 20 mg/L.

The purpose of CT process is to retain and consume the MFT with the coarse solids to eventually create a sustainable and acceptable dry or wet landscape (MacKinnon *et al.* 2000). Technologies like CT give the oil sands

companies the ability to create broadly diverse landscapes that help fulfill the commitment to the reclamation of the disturbed areas.

*2.3.4 The options for fine tailings management*

The fine tailings management and reclamation programme must ensure that (FTFC 1995): (i) the direct contact of fine tailings with the environment and the release of contaminants into the environment are restricted; (ii) the reclaimed area is stable, productive, and biologically self-sustaining.

To date, no single reclamation option can handle the huge volumes of fine tailings in a technically, environmentally, and economically acceptable manner. The reclamation of the fine tailings requires an integrated approach. The reclamation of the fine tailings will be accomplished through a combination of so called "dry" and "wet" landscape techniques (FTFC 1995).

The dry approach involves dewatering the fine tailings or incorporation of the fine tailings with a solids material and being reclaimed as a land surface. Some of the various dry landscape options include: (i) dewatering of the fine tailings through processes such as evaporation and freeze-thaw; (ii) incorporation of fine tailings with overburden clays; and (iii) creating NST or CT (FTFC 1995).

The wet approach involves a lake system, whereby the fine tailings are capped with a layer of water to be isolated from direct contact with the surrounding environment (FTFC 1995). The wet landscape option disposes of

MFT as a fluid or non-trafficable deposit. The objective of this reclamation option is to produce a stable, productive, and self-sustaining cap water zone, which can support viable aquatic ecosystems shortly after water capping and is free of long-term maintenance. The surface mining practice of the oil sands industry leaves large mined-out pits, which may be used as the geotechnically secure areas for long-term containment of the fluid fine tailings (FTFC 1995).

Each approach in fine tailings management has its advantages and disadvantages. The final strategy will depend on detailed technical and environmental assessments, economic consideration, and the stakeholder's acceptance (FTFC 1995).

## 2.4 SRB and methanogens

### *2.4.1 The SRB*

SRB are a diverse group of strictly anaerobic bacteria that share the ability to use sulphate as the terminal electron acceptor and conduct dissimilatory sulphate reduction in the oxidation of organic matters. In the dissimilatory sulphate reduction, sulphate is reduced to sulphide according to Equation 2-2 (Levett 1991):

$$CH_2O + SO_4^{2-} \longrightarrow HCO_3^- + S^{2-} \quad \text{.....(2-2)}$$

where $CH_2O$ represents the organic substrates.

The substrates used by SRB include lactate, pyruvate, and acetate, among many others. Lactate and pyruvate are all converted to acetate and $CO_2$ as the major end products by acetate-producing organisms. In the acetate-utilising species, $CO_2$ is a major product (Postgate 1984).

Sulphide is a normal metabolic product of dissimilatory sulphate reduction and can render soluble iron insoluble by converting it to FeS. Thus, the SRB colonies can be identified by the precipitation of black FeS (Levett 1991).

SRB are adaptable to almost any anaerobic environment. SRB need anaerobic and reducing conditions for growth. The redox potential (Eh) of the growth media should be around –100 mV (Levett 1991; Postgate 1984). However, some SRB can survive long exposure to oxygen and become active again if the environment becomes anaerobic. SRB can tolerate temperatures from –5 to +75 °C and pH values ranging from 5 to 9.5 (Postgate 1984).

The activity of SRB in a given environment will change the chemical and physical nature of that environment. Due to the release of $CO_2$ and accumulation of carbonate and bicarbonate, the aqueous environment will have elevated alkalinity and metal ions will precipitate as insoluble metal sulphides (e.g. FeS). If no metals are present or pH is low enough, the sulphide could be converted to hydrogen sulphide ($H_2S$) and escape into the water phase or the atmosphere (Gray 1989). Sulphate reduction will consume a substantial amount of organic substrates and produce, sometimes via acetate, $CO_2$ (Postgate 1984).

Given a high ratio of metabolisable carbon to sulphate, SRB can deplete an environment of sulphate almost completely (Postgate 1984).

Sulphite and thiosulphate are intermediates in the normal sulphate reduction process, and can also be used as substitute electron acceptors for the growth and carbon metabolism of some SRB species (Levett 1991; Postgate 1984).

The disappearance of sulphate in nature over time may be used as an index of the SRB activity (Levett 1991; Postgate 1984).

### *2.4.2 The methanogens*

Methanogens are a morphologically diverse group of anaerobic bacteria unified by their ability to produce methane ($CH_4$) (Levett 1991).

Methanogens utilise a limited number of simple carbon compounds as carbon and energy sources for methanogenesis, including $CO_2$, formate, methanol, and acetate (Levett 1991). More than half of the biogenic methane in nature is believed to originate from acetate (Postgate 1984). Methanogens are usually considered to be the last players in the decomposition of organic matters in anaerobic ecosystems. That is, they consume the metabolic end products (mainly acetate and $H_2$) of other strictly anaerobic bacteria as energy sources and produce $CH_4$ as their waste product. $CO_2$ and $HCO_3^-$ serve as the terminal electron acceptors during anaerobic respiration by methanogens (Fedorak *et al.* 2000).

Methanogens are found in a variety of anaerobic habitats including sediments, sludge, and animal waste digesters. They require an Eh in the range of –150 to –220 mV to thrive (Fedorak *et al.* 2000). In general, methanogens get inactivated by the presence of oxygen, although not every species is rapidly killed by oxygen (Levett 1991).

The most common method used to enumerate specific types of methanogens is the most probable number (MPN) technique, where the sample is serially diluted and inoculated into a suitable broth medium with a specific substrate (Levett 1991).

Methane, being a gas, diffuses from anaerobic zones and can also become subject to aerobic mineralisation by methanotrophic bacteria (Postgate 1984).

### *2.4.3 The competition between SRB and methanogens*

SRB have long been known to be incompatible with methanogens, although both of them are strict anaerobes. Indeed, they seem in nature to have a somewhat competitive relationship. The methanogens take over when the SRB have done what they can (Postgate 1984).

A widely accepted view about the incompatibility of methanogenesis and sulphate reduction by SRB suggests that the SRB can out-compete methanogens for common substrates, $H_2$ and acetate, except in some particular environments with sulphate deficiency (Postgate 1984). It has long been known that methane production in marine sediments occurs only after sulphate has been depleted

from the pore water. This is due to the competition between methanogens and SRB for some electron donors. Based on thermodynamic considerations, the utilisation of $H_2$ or acetate by SRB yields more energy than the utilisation of these substrates by methanogens. When sulphate is depleted, methanogens carry out the terminal steps in the anaerobic environment (Fedorak *et al.* 2000). In most environments there is little or no overlap between the zone of methanogenesis and the zone of sulphate reduction by SRB in sediments. Methanogenesis begins when sulphate is depleted (Schlesinger 1997).

Like methanogens, SRB can also use acetate and $H_2$ as energy sources. However, SRB use a wider range of organic compounds as their energy sources compared to methanogens and produce sulphide as their waste product (Fedorak *et al.* 2000).

Sediments in which sulphate reduction is active often lie over sediments in which methane production is taking place. Methane can be a substrate for sulphate reduction by SRB as shown in Equation 2-3 (Schlesinger 1997; Postgate 1984):

$$CH_4 + SO_4^{2-} \longrightarrow HS^- + HCO_3^- + H_2O \quad \text{.....(2-3)}$$

Acetate is known to be a good substrate for methane formation. Therefore, the product of sulphate reduction by the acetate-producing organisms would be expected to favour methanogenesis, indicating the complex relationship between SRB and methanogens (Postgate 1984).

# 3.0 Problem statement and the objective of this project

## 3.1 Problem statement

As discussed in Section 2.3, as a tailings management option in the oil sands industry, CT is a type of NST and is made up of coarse sands, MFT, and gypsum. Upon deposition, the CT slurry releases its porewater quickly and the deposit becomes denser. The released water will be recycled as the process water.

The integration of CT into tailings disposal and management will affect the water properties in the oil sands operation. The CT release water must be suitable for recycle and not limit reclamation options. The addition of chemical coagulants in CT process can lead to the direct shifts in pH, alkalinity, salinity, as well as the cations and anions associated with the coagulants (MacKinnon *et al.* 2000). In addition, changes in the water quality will occur after deposition through exchanges and interactions with the atmosphere and clays, and bio-chemical reactions. Some of the possible water quality issues associated with the use of CT process are increase in salinity, scaling potential from increased $Ca^{2+}$ content, change in pH, corrosion potential, potential effect on bitumen recovery, and impact on reclamation (MacKinnon *et al.* 2000).

In the gypsum-treated CT mixture, the main water quality change is the increase in $Ca^{2+}$ and $SO_4^{2-}$ contents. The CT release water will have $Ca^{2+}$ content in the range of 70 to 120 mg/L and $SO_4^{2-}$ content more than 1000 mg/L (MacKinnon *et al.* 2000). Excess $Ca^{2+}$ in the recycle water can decrease bitumen recovery (FTFC 1995; Kasperski 1992). Bitumen recovery will be significantly

affected when the calcium concentration exceeds 3 mM (i.e. 120 mg/L) (FTFC 1995). The increased $Ca^{2+}$ content will also raise issues regarding scaling and corrosion in the process units (MacKinnon *et al.* 2000). The elevated concentration of $SO_4^{2-}$ from CT in the recycle water stream may also pose adverse effects on bitumen recovery (FTFC 1995). The high concentration of $SO_4^{2-}$ content in the CT water is the main factor causing the increase in salinity. In studies on potential terrestrial impacts of CT water on vegetation, it has been shown that the salinity is the major factor that needs to be addressed (MacKinnon *et al.* 2000).

At Syncrude Canada Ltd., a preliminary pilot-scale field test (Shaw *et al.* 1996) was carried out from 1995 to 2000 for studying whether the co-disposal of CT placed beneath MFT is a feasible disposal technology. The main objectives of that test were to determine if CT would flow under MFT without segregation, if CT would dewater under MFT and the dewatering rate, and if and how the CT release water would affect the physical and chemical properties of the overlying MFT and cap water.

The main findings of this field demonstration test were as follows (Shaw *et al.* 2001; Shaw *et al.* 1996).

(i) The CT and MFT layers maintained their distinct characters during and after the placement of fresh CT slurry below a lay of MFT.

(ii) The CT layer densified with its solids content increasing from approximately 60 wt% to 78 wt% during the period from 1995 to 1999.

(iii) Enhanced densification through dewatering was observed in the MFT layer with the solids content increasing from 33 wt% to 41 wt% during the study period.

(iv) The produced surface water (i.e. cap water) showed a decrease in conductivity and in particular in $SO_4^{2-}$ and $Ca^{2+}$ concentrations, most likely due to the microbiological activities, dilution of the CT release water with the displaced MFT porewater, or precipitation reactions.

The field demonstration test indicates that the co-disposal of CT beneath MFT deposition initiates beneficial changes for tailings disposal with respect to the release water quality and both the MFT and CT densification rates. However, prior to recommending this co-disposal scheme as a viable full-scale tailings management alternative, it is necessary, under controlled laboratory conditions, to understand the physical, chemical, and microbiological processes that will occur in this deposit.

### 3.2 Objective of this project

The overall objective of this project is to demonstrate whether the co-disposal of CT beneath MFT is a feasible technology in tailings disposal and management under controlled experimental conditions, with respect to the cap water quality for recycle and the densification rates of both the MFT and CT layers. More specifically the objective is to:

(i) determine the effects of this CT beneath MFT deposition scheme on the produced cap water quality, mainly in terms of hardness ($Ca^{2+}$ + $Mg^{2+}$) and $SO_4^{2-}$ concentration in the cap water;

(ii) observe the physical, chemical, and microbiological changes that occur in the MFT zone under anaerobic conditions as the CT release water interacts in the MFT layer, including the sinks of $SO_4^{2-}$ and $Ca^{2+}$, and the densification rate of MFT;

(iii) observe the physical, chemical, and microbiological changes that occur in the CT layer under anaerobic conditions, including the sinks of $SO_4^{2-}$ and $Ca^{2+}$, and the densification rate of CT.

As the CT release water, rich in $Ca^{2+}$ and $SO_4^{2-}$ ions, percolates through the MFT layer, it is expected that the $Ca^{2+}$ may be exchanged and sequestered on MFT clay surface, while the $SO_4^{2-}$, through microbiological activity, will be converted into sulphide, particularly iron sulphide. Lowered $Ca^{2+}$ and $SO_4^{2-}$ contents in the release water of this co-disposal deposit will be beneficial for re-using the water in oil sands extraction process. Increased densification rates of both the MFT and CT will also be beneficial in reducing the volumes that these deposits occupy in the tailings ponds.

# 4.0 Methods and materials

## 4.1 Methodology

This research used static column mesocosms to study and monitor the physical, chemical, and microbiological changes occurring in this CT beneath MFT co-disposal system, as detailed below.

The static column mesocosms consisted of four different deposit systems, as described in Table 4-1.

**Table 4-1. Static column mesocosm systems.**

| System | Column configuration during incubation | Deposit at column filling | Number of columns |
|---|---|---|---|
| 1 | Cap water/MFT/CT | A 27-cm height of MFT on the top of a 54-cm height of CT. | 20 |
| 2 | Cap water/MFT/CT | A 27-cm height of MFT on the top of a 54-cm height of CT. | 6 |
| 3 | Cap water/MFT | Only a 54-cm height of MFT. | 12 |
| 4 | Cap water/CT | Only a 54-cm height of CT. | 12 |

System 1 was the main system to be studied. Systems 3 and 4 served as controls for System 1. At column filling, System 1 contained a 27-cm height of MFT layer on the top of a 54-cm height of CT layer, while Systems 3 and 4 contained only a 54-cm height of MFT or CT layer. No extra water was added to the cap of the columns at column filling. All the cap water produced during the ensuing incubation was the released water from the deposit.

For Systems 1, 3, and 4, the columns were incubated for a maximum of 1 y under dark and temperature controlled conditions. After a certain incubation period, the columns were sacrificially sampled. For each column at its sacrificial

sampling, the column headspace gas, cap water, and MFT or CT samples were analysed. The analyses might include:

(i) the column headspace gas analysis for $CH_4$, $O_2$, $N_2$, $CO_2$, $H_2S$, and $C_2H_4$;

(ii) water (cap water and MFT or CT porewater) analysis for pH, electrical conductivity (EC), $HCO_3^-$, major cations of $Ca^{2+}$, $Mg^{2+}$, $Na^+$, $K^+$, $NH_4^+$, and major anions of $SO_4^{2-}$, $S^{2-}$, $PO_4^{3-}$, $NO_3^-$, $NO_2^-$, $Cl^-$;

(iii) MFT or CT analysis for trapped gases of $CH_4$, $O_2$, $N_2$, $CO_2$, $H_2S$, and $C_2H_4$, redox potential, the contents of oil, water, and solids (O/W/S), particle size distribution, acid volatile sulphides (AVS), and the quantification of microbial populations of methanogens and SRB using MPN method.

Also, the volume changes of the various zones (cap water, MFT, or CT) and the dewatering rates of MFT and CT were followed by visually recording the changes in the interface positions of cap water and MFT (in Systems 1 and 3), cap water and CT (in System 4), or MFT and CT (in System 1). For System 1, the interface position of MFT and CT was measured only at the time of sacrificial column sampling.

System 2 had the same deposit configuration as System 1, but it was designed in particular to regularly conduct headspace gas and cap water sampling and analysis, and to regularly record the position change in the interface of MFT and CT in an in-situ manner without sacrificing the columns. Furthermore, the columns in System 2 were sacrificially sampled only at the end of 1 y of incubation.

The details of the design and analysis for these static column Systems 1, 2, 3, and 4 are presented in the following protocol development Section 4.2.

## 4.2 Protocol development

Previous research (Fedorak *et al.* 2000) on oil sands tailings in the Department of Biological Sciences, at the University of Alberta, involved extensive physical, chemical, and microbiological investigations of the MFT and CT materials using a variety of innovative experimental methods. Fedorak *et al.* (2000) also developed and adopted many proven protocols for column setup and analyses to evaluate the CT and MFT under anaerobic conditions. In developing the protocols for this current project, the protocols used by Fedorak *et al.* (2000) were frequently referenced.

### *4.2.1 Number of columns in static column mesocosms*

The planned incubation time was 1 y. The number of columns for Systems 1, 3, and 4 was determined to allow for a minimum of one column to be sacrificially sampled every month in each system. For System 2, no sacrificial sampling was designed until the end of the planned incubation periods, and therefore a reasonable number of columns were chosen. Table 4-1 shows the number of columns used in each system.

*4.2.2 Column design for Systems 1, 2, 3, and 4*

Systems 1, 2, 3, and 4 used a "12.1 ID × 12.7 OD × 91.4 H" cm clear acrylic column as their mesocosms. The top of the columns was covered with a 15×15×6 mm square acrylic plate, and the bottom of the columns was covered with a 22×22×12 mm square acrylic plate.

The fabrication of the pieces needed for assembly of the static columns was completed in the Geotechnical Engineering Laboratory in the Department of Civil and Environmental Engineering, at the University of Alberta. The pre-fabricated acrylic pieces were bonded using "Weld-On 16#" acrylic glue (from Johnston Industrial Plastics, Edmonton, Alberta) and assembled in Environmental Engineering Laboratory, University of Alberta, at the time of column filling. The designs of the column pieces are detailed below.

4.2.2.1 Design of the top covers

For Systems 1, 3, and 4, there were 2 holes placed in the top cover. A 3.2-mm NPT threaded hole was used for connecting a 1-L Tedlar bag (catalog Number 231-01, purchased from Safety Instruments Ltd., Edmonton, Alberta) through a 25-cm length of 3.2 ID × 6.4 OD mm Tygon tubing to collect the evolved headspace gas in the columns. A 10-mm diameter straight hole was used for fitting a rubber septa, which would allow for cap water sampling in an in-situ manner by using syringe if needed.

For System 2, there were four holes placed in the top cover. A 3.2-mm NPT threaded hole was used for connecting a 1-L Tedlar bag through a 25-cm

length of 3.2 ID × 6.4 OD mm Tygon tubing to collect the evolved headspace gas in the columns. A 3.2-mm NPT threaded hole was used for fitting a 3.2-mm OD stainless steel tubing to regularly monitor the position change in the interface of MFT and CT in an in-situ manner. A 3.2-mm NPT threaded hole was used for fitting a 3.2-mm OD stainless steel tubing to regularly sample the cap water in an in-situ manner. A 1.6-mm NPT threaded hole was used for fitting a 40-cm length of 1.6-mm OD stainless steel tubing to regularly conduct the head space gas analysis by gas chromatography (GC) in an in-situ manner.

4.2.2.2 Design of the bottom covers

For Systems 1 and 2, there were two 1.9-cm diameter straight holes placed in the bottom cover. One hole was used to finally fill the column with CT through a funnel, and the other hole was used to exit the trapped air before sealing the bottom cover. The two holes were plugged at the end of column filling using 1.9 mm diameter acrylic rods. For Systems 3 and 4, the bottom cover did not contain any hole design.

4.2.2.3 Reinforcement of the seals

Several triangular pieces of acrylic (two for the top cover and four for the bottom cover) were used to reinforce the seals between the column and covers.

#### 4.2.2.4 Column stopper for Systems 1 and 2

Systems 1 and 2 contained a layer of MFT on the top of a layer of CT. Because of the special column filling procedures in these two systems, a column stopper was used to fill the columns of Systems 1 and 2, as detailed in the following Section 4.2.4.

#### 4.2.2.5 Monitoring the interface position of MFT and CT in Systems 1 and 2

The major purpose of monitoring the interface position between MFT and CT layers in Systems 1 and 2 was to determine the densification rates of individual MFT and CT layers in these co-disposal deposition systems. To monitor the position change in the interface of MFT and CT in Systems 1 and 2, a piece of Geo-grid with a mesh hole size of approximately 0.5 × 0.5 cm (from Nilex Inc. Edmonton, Alberta) was placed at the interface of MFT and CT during column filling. The Geo-grid piece physically separates the MFT and CT layers, but does not interfere with the transport of the CT release water. Thus, by monitoring the position of the Geo-grid piece, the position change in the interface of MFT and CT could be obtained. For System 1, the Geo-grid position was only measured at the time of sacrificial column sampling. For System 2, the Geo-grid position was regularly monitored by using a stainless steel tubing in an in-situ manner. The stainless steel tubing was to be contacted to the Geo-grid piece at the time of measuring the Geo-grid position.

*4.2.3 The MFT and CT materials*

About 800 kg each of CT and MFT slurries were delivered in 20-L plastic pails from Syncrude Canada Ltd. in December 2001. Both the CT and MFT were kept in a 4°C temperature controlled room until use.

At the time of column fillings, in order to reduce the heterogeneity of the CT and MFT materials among different columns, several pails of the MFT or CT materials were randomly selected and well mixed in containers of suitable size. The mixed MFT or CT materials were then used to fill the columns.

*4.2.4 Procedures for column filling and assembly in Systems 1, 2, 3, and 4*

Prior to filling column with MFT and CT, each column was flushed with $N_2$ for 2 to 3 min. $N_2$ flushing continued during column filling. After column filling, the column headspace was flushed with $N_2$ for 2 to 3 min. The top cover of the columns was then bonded to the column using "Weld-On 16#" acrylic glue and temporarily clamped together to ensure a good seal. The 1-L Tedlar bag was flushed with $N_2$ three times prior to its connection to the column cover.

In the columns of Systems 1 and 2, both MFT and CT were to be layered in the same column, with a 27-cm height of MFT layer placed above a 54-cm height of CT layer. Due to the characteristics of the MFT and CT materials, the column filling for Systems 1 and 2 needed a special procedure.

In developing the protocol for column filling in Systems 1 and 2, two issues were considered. First, if the CT is placed first in the column, the CT will

start releasing its porewater quickly (in several hours after filling) and this porewater will accumulate at the surface of CT before placing the MFT in the column. Second, the freshly mixed MFT will have a very different structure from that of the field MFT, and thus some "maturing" of the MFT will be needed before being deposited above the CT. Considering these two factors, and after consultation with the industrial partner of this project, the column filling for Systems 1 and 2 used a procedure that involves filling the column with MFT first, letting the MFT "mature" for some time, then filling the column with CT, and finally inverting and sealing the column. This procedure required the column to be inverted at the time of filling with MFT and CT. A column stopper was designed for this purpose. Figure 4-1 shows the schematics of the side view of the column stopper as it stands inside the column at column filling.

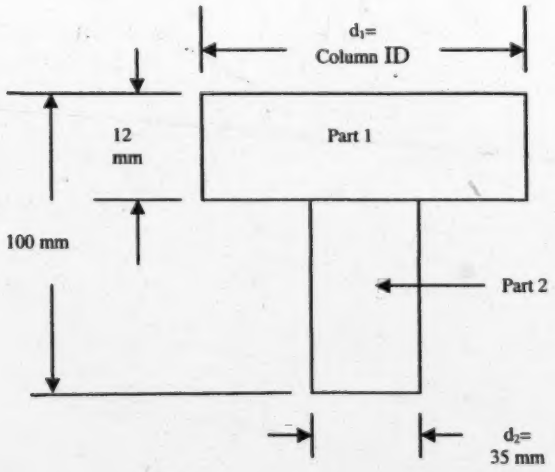



**Figure 4-1. Schematics of side view of the column stopper used for filling columns in Systems 1 and 2** (not to scale)

The column stopper had a suitable external diameter, which was close to the column ID (inside diameter). Thus, the column stopper could minimise the MFT leaking during filling column with MFT and CT and be removed relatively easily after column filling. Four throughout holes of about 8 mm diameter were also designed in Part 1 of the column stopper to ease the removal of the column stopper after column filling. The four holes were stopped using nuts during column filling. The total height of the column stopper was approximately 10 cm. This 10 cm height of column stopper would create a 10 cm height of headspace after the column filling and stopper removal.

The protocol for column filling for Systems 1 and 2 can therefore be detailed as follows (see Figure 4-2).

(i) The column stopper was first installed onto one end of the column, and the column was filled with a 27-cm height of freshly mixed MFT under a $N_2$ atmosphere (see Figure 4-2a). After flushing the headspace of these MFT-filled columns with $N_2$ for a few minutes, the columns were temporarily covered with a plastic film to maintain the $N_2$ atmosphere in the column headspace. The MFT in the column was allowed to "mature" overnight to develop some structure, similar to that seen in the field.

(ii) On the second day, a piece of Geo-grid was first placed on the top of the MFT layer. The Geo-grid piece had a diameter close to the column ID. The purpose of using this Geo-grid is to physically separate the MFT and CT layers and provide a method to monitor the position change in the interface between

the MFT and CT during the ensuing incubation. After placing the Geo-grid, a 54-cm height of freshly mixed CT was placed on the top of MFT (see Figure 4-2b). Special caution was used when filling with CT to minimise the impact of the CT flow on the underlying MFT and to avoid disturbing or displacing the Geo-grid. After sufficient amount of CT was placed in the column, the acrylic bottom cover with two 1.9-cm diameter straight holes was used to seal the CT side of the column end, using "Weld-On #16" glue. The two 1.9-cm diameter straight holes were used for final column filling with funnel and to allow the trapped air to escape. The two holes were plugged using 1.9-cm diameter acrylic rods at the end of column filling with CT.

(iii) After filling with CT, the column was turned over, so that the CT layer was at the bottom and the MFT layer was at the top. The column stopper was then removed, creating a 10-cm height of headspace. The headspace was then flushed with $N_2$ for several minutes and the column was finally sealed with the acrylic top cover of suitable design (see Figure 4-2c).

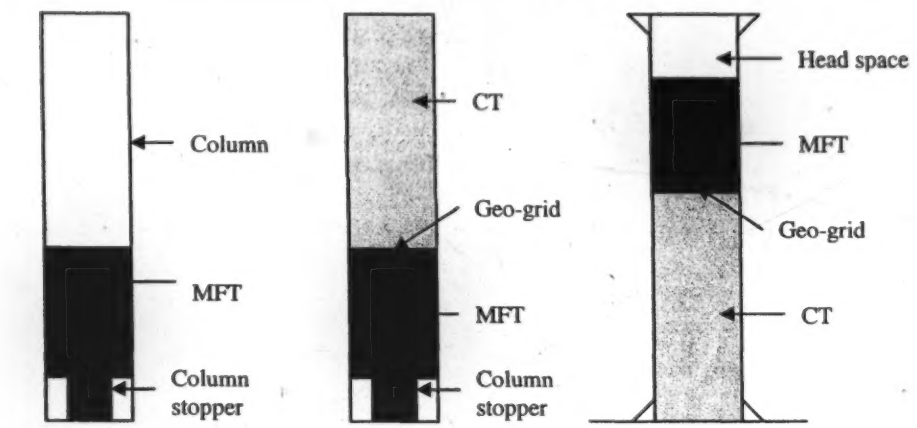



**Figure 4-2. Schematics of column filling procedures for Systems 1 and 2** (not to scale)

*4.2.5 Incubation and regular monitoring of the columns*

After filling, all static columns were moved to the incubation room. The columns were maintained at 18 to 20°C, under dark conditions during incubation.

For all columns in Systems 1, 2, 3, and 4, at suitable intervals of several days at the beginning of incubation to longer periods at later incubation, the height of the produced cap water and the interface position between the cap water and MFT or cap water and CT layers were measured. These data would be used to determine the water release rate of the deposit and the MFT and/or CT

densification rates. Gas bubble evolution and other possible visual changes in the MFT and CT layers were also recorded.

For System 2, the columns were not sacrificially sampled during the incubation period. At suitable time intervals, the cap water extraction, Geo-grid position monitoring, and headspace gas analysis by GC were regularly performed in an in-situ manner. The order for these analyses was to first conduct the Geo-grid position recording, then conduct the headspace gas GC analysis, and finally perform the cap water extraction under $N_2$ atmosphere. Particular attention was paid to the cap water extraction. The cap water was extracted using a 60-mL syringe through the 3.2-mm OD stainless steel tubing, which was fixed to the top cover. At every cap water extraction, the existing cap water was completely removed. This complete removal of the cap water sample ensures that every time the sampled cap water represents only the water released since the last sampling. This practice can produce a time series of cap water composition change. The void volume created in the column headspace by the removal of cap water was filled with $N_2$ gas. Because an adequate volume of cap water was needed at every sampling, the sampling intervals for System 2 were based on the volume of produced cap water and were short at the beginning of incubation and long at the later incubation.

The Tedlar bags were checked regularly. If the Tedlar bag was fully filled with evolved gas, the existing Tedlar bag would be replaced with an empty Tedlar bag. The GC analysis and gas volume measurement of the Tedlar bag gas were conducted for every removed Tedar bag.

*4.2.6 Sacrificing and freezing procedures of the columns*

At the end of the planned incubation period, the columns were sacrificed for sampling and analysis. Prior to sacrificing the column, the Tygon tubing (which connected the Tedlar bag to the column top cover) was first clamped and the Tedlar bag was removed for gas analysis by GC and gas volume measurement using a 60-mL syringe (from Labcor Inc., Anjou, QC). Then, under a $N_2$ atmosphere, the top cover of the column was removed and the cap water was sampled.

For System 1, the microbial MPN analyses of SRB and methanogens were conducted at a frequency of usually once every 60 d. The sampling for MPN analysis was completed before the column freezing. The sampling used a core-sampling method (with a 6.4-mm diameter hole) under a $N_2$ atmosphere. The samples for MPN analysis were taken from the centre of the column using a stretch of suitable size rigid plastic tubing. The samples were then transported in a 60-mL syringe or a 90-mL plastic jar (made of clarified polypropylene) filled with $N_2$ to the Biological Sciences Laboratory at the University of Alberta. All materials used for MPN sampling (plastic tubing, syringe, and jar) were autoclaved at 121°C for 20 min prior to use.

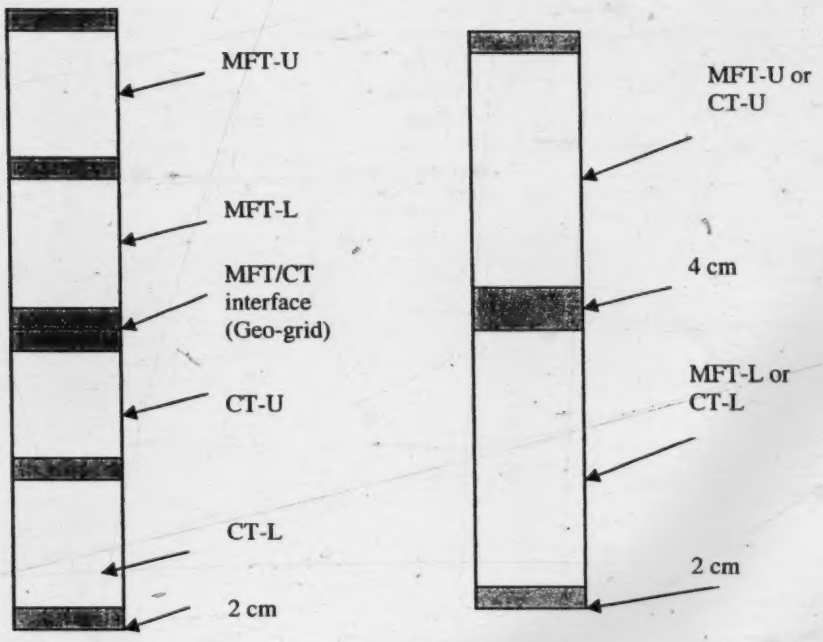
For MFT, two samples were taken for MPN analysis. One sample was taken from approximately 4 cm below the interface of the cap water and MFT, and the other was taken from approximately 4 cm above the MFT and CT

interface, which was indicated by the position of the Geo-grid. For CT, only one sample was used for MPN analysis. This sample was taken from the middle of the CT layer.

The columns in Systems 1, 3, and 4 were sacrificed at a required interval. After the samplings of Tedlar bag gas, cap water, and MPN samples, the columns were placed in a freezer at a temperature of about –20°C and left for several days to reach a complete frozen state. The columns in System 2 were sacrificed only at the end of 1 y of incubation.

*4.2.7 Frozen column cutting and sampling*

Figure 4-3 shows the scheme of frozen column cuttings for Systems 1, 2, 3, and 4, with the shaded areas to be cut off.




(a). For Systems 1 and 2 (b). For Systems 3 and 4

**Figure 4-3. Scheme of frozen column cuttings with the shaded areas to be cut off** (not to scale)

4.2.7.1 Frozen column cutting and sampling for columns in Systems 1 and 2

As shown in Figure 4-3a, two MFT and two CT samples from the upper and lower zones of MFT or CT were equally taken in Systems 1 and 2, using the following procedures. First, cut at the Geo-grid position to separate the MFT and CT layers. Then cut off 2 cm from either side in both MFT and CT layers. Further, cut off 2 cm in the middle of the remaining MFT and CT layers to

separate the individual MFT and CT into two samples, respectively. The two MFT and two CT samples were denoted as MFT-U, MFT-L, and CT-U, CT-L, respectively, where U refers to samples from the upper half zone and L refers to samples from the lower half zone of the MFT or CT layer.

4.2.7.2 Frozen column cutting and sampling for columns in Systems 3 and 4

As shown in Figure 4-3b, two MFT or CT samples from the upper and lower zones of MFT or CT were equally taken in Systems 3 and 4, using the following procedures. First cut off the MFT or CT in the middle to separate the MFT or CT into two equal parts. Then cut off 2 cm from either side of the MFT or CT layer to produce two MFT or CT samples. The two MFT or CT samples were denoted as MFT-U, MFT-L, and CT-U, CT-L, respectively, where U refers to samples from the upper half zone and L refers to samples from the lower half zone of the MFT or CT layer.

*4.2.8 Subsampling of frozen MFT and CT*

The frozen MFT or CT samples were brought to room temperature in 4-L glass jars (filled with $N_2$) overnight. Then, subsamplings were performed on the MFT or CT samples in a $N_2$ chamber. The following samples were prepared.

The porewater extraction was completed using a combination of centrifugation and filtration procedures. For centrifugation of the MFT and CT samples, a Sorvall RC-5B centrifuge with SS-34 rotor and eight Teflon tubes of

50 mL each was used at a relative centrifugal force of 12,100 g for 30 min. Immediately after the centrifugation, pre-filtration using AP 15 Millipore glass fiber filter and vacuum filtration under a $N_2$ atmosphere was performed to remove coarse solids and bitumen. A second filtration was conducted using a disposable 0.45 μm Millipore Millex syringe filter (Millipore, Billerica, Massachusetts, USA).

The MFT and CT subsamples for trapped gas, redox potential, oil/water/solids contents, particle size distribution, and AVS analysis, among others, were also taken at this time under a $N_2$ atmosphere. These subsamplings were conducted by first homogenizing the MFT or CT under $N_2$ atmosphere.

*4.2.9 Eh measurement on MFT and CT samples*

The instrument used for Eh measurement was an Accumet Model 50 pH/Ion/Conductivity Meter with a Cole-Parmer Ag/AgCl ORP electrode. The ORP electrode was calibrated against a standard ORP solution (from Orion Research Inc. Beverly, MA, U.S.A.) both before and after the Eh measurements.

For Eh measurement, the MFT or CT samples were contained in a 120-mL glass jar. During the Eh measurement, the headspace of the jar was filled with $N_2$. In some samples, the Eh would require a long time to reach a stable point. In order to obtain accurate and consistent results, the Eh readings after 1 and 2 h of measuring time were recorded. The Eh readings at 2 h of measuring time were used for data analysis.

*4.2.10 Sulphide analysis in water sample*

The $S^{2-}$ analysis in the cap water and porewater samples was performed using "CHEMetrics sulfide test kit" (from CHEMetrics Inc., Calverton, Virginia, U.S.A.). The CHEMetrics test kit employs a methylene blue colorimetric method, and will measure total acid soluble sulphides, including dissolved $H_2S$, $HS^-$, and acid-soluble metallic sulphides in suspension. Results are expressed as mg of S/L.

Based on the previous sulphide analysis results in MFT and CT porewaters (Fedorak *et al.* 2000), a sulphide test kit with two ranges of 0 to 1 and 1 to 10 mg/L each (catalogue number K-9510) was used. The minimum detection limit of this test kit was 0.05 mg/L.

The water samples for $S^{2-}$ analysis were first filtered using a 0.45-μm syringe filter. To avoid or minimise the oxidation of sulphides, water samples were prepared with minimum aeration under a $N_2$ atmosphere and were analysed immediately after the preparation.

*4.2.11 MPN analysis on MFT and CT samples*

Enumeration of viable microorganisms was accomplished by MPN technique. Only SRB and methanogens were enumerated as these are the most important groups of microorganisms in MFT and CT deposits (Fedorak *et al.* 2000).

The MPN analyses of SRB and methanogens on the MFT and CT samples were conducted using the 3-tube MPN method (Fedorak *et al.* 2000). Due to the laborious nature of the MPN analysis work, the MPN analyses were conducted only on the MFT and CT samples from System 1 at selected incubation times.

The result of MPN analysis used a unit of "MPN/g (of dry solids)" for the MFT or CT. Because the MPN samples for MPN analysis were dispensed as wet materials, the ratio of dry weight to wet weight of the CT and MFT samples was needed for the unit conversion. This ratio was determined in the Environmental Engineering Laboratory by drying the MFT and CT samples in a 104°C (103 to 105°C) oven overnight, following the procedures discussed in APHA (1995). The MFT and CT samples for this drying analysis were collected at the time of taking the MPN samples, using suitable glass containers. The weights of the MFT and CT samples were measured before and after the drying, and the ratio was then calculated.

### *4.2.12 Trapped gas analysis on MFT and CT samples*

The term "trapped gas" in this thesis was used to describe the gas extracted from the MFT or CT samples. Thus, the trapped gas may include both trapped and dissolved gases in the MFT and CT. In this project, the purpose of the trapped gas analysis on MFT and CT was to qualitatively determine if $CH_4$ (and possibly $H_2S$ and $C_2H_4$) was (or were) present in the MFT and CT. Due to

the high cost of trapped gas analysis on oil sands tailings in the commercial laboratory, with the consent of the industrial partner of this project, the trapped gas analyses of MFT and CT were completed in the Environmental Engineering Laboratory using a self-developed gas extraction and GC analysis method.

The aim of gas extraction method development for trapped gas was to maximise the release of the trapped gas. The following method was developed after some preliminary tests.

(i) In a 160-mL serum bottle, first placed 60 mL of MFT or CT sample, and then filled the serum bottle with 80 mL of $N_2$-degassed (degassing for about 1 h) deionised water.

(ii) Flushed the headspace of the serum bottle with helium (He) gas for a few minutes and then closed the bottle with a rubber septum. Shook the serum bottle thoroughly for 2 min using a wrist-action shaker.

(iii) Let the serum bottle sit overnight to reach equilibrium between the headspace gas and the slurried sample.

(iv) Using the GC to perform the headspace gas analysis in the serum bottle.

*4.2.13 Physical and chemical analyses on water, MFT, and CT samples*

These analyses include pH, EC, alkalinity, cations, anions, selected trace elements, residual bitumen, solids content, and particle size distribution. These analyses were performed by Syncrude Analytical Group at the Edmonton

Research Centre, using established company protocols and standard methods (Fedorak *et al.* 2000).

*4.2.14 Gas analysis by GC*

From Fedorak *et al.* (2000), the main gases detected in the headspace of column mesocosms containing MFT or CT deposit and the gases trapped in the MFT and CT were $O_2$, $N_2$, $CH_4$, and $CO_2$. In addition, some low quantities of $H_2S$ and $C_2H_4$ might be present. Therefore, in this work, the GC analyses for Tedlar bag gas in Systems 1, 3, and 4, for in-situ column headspace gas in System 2, and for trapped gas in MFT and CT were focused on quantitatively measuring the $O_2$, $N_2$, $CH_4$, $CO_2$, $H_2S$, and $C_2H_4$.

A Varian CP-2003 portable Micro-GC with two channels of A and B was used for all the gas analyses. Channel A was installed with the CP-Mole Sieve column and a TCD detector. Channel A is suitable for the analyses of $H_2$, $O_2$, $N_2$, and $CH_4$. Channel B was installed with the HayeSep column and a TCD detector. Channel B is suitable for the analyses of $CH_4$, $CO_2$, $C_2H_4$, and $H_2S$. He was the carrier gas for both columns.

To maintain the Mole Seive column (in channel A) and HayeSep column (in channel B) in good working conditions, the GC columns were regularly "conditioned" at 170°C for Mole Seive column and at 140°C for HayeSep column for 2 to 4 h.

The Micro-GC instrument had an internal sampling pump to automatically retrieve the gas samples. Therefore, no syringe injection was needed for the GC sampling. The container of the gas sample (e.g. the Tedlar bag) was directly connected to the GC sampling port. A suitable sampling time was set to flush out the possible deadspace volume on the gas route between the GC and the gas sample container before the sample injection. The sample injection time was set to be 40 ms. This injection time results in an average injection volume of 200 nL (VCI 1999).

The flowrate reading of the carrier gas has to be measured at its flow-out ports at the back of the Micro-GC. This flowrate was too small to be measured using the available flowrate measuring instrument in this study. To keep the constant flowrate of the carrier gas, the GC was always connected to the carrier gas cylinder with constant 552 kPa outlet pressure. Thus, when running the GC, the carrier gas was always on and at constant flowrate.

Table 4-2 summarises the main parameters of the Micro-GC for GC calibration, Tedlar bag gas analysis in Systems 1, 3, and 4, in-situ column headspace gas analysis in System 2, and trapped gas analysis in MFT and CT.

**Table 4-2. The main parameters for the Micro-GC gas analysis.**

| Channel name | TCD A | TCD B |
|---|---|---|
| Channel preference | Active | Active |
| Column preference | Hi temp. | Lo temp. |
| Column temperature (°C) | 110 | 50 |
| TCD offset | On | On |
| Sensitivity | Lo | Mid |
| Injection time (ms) | 40 | 40 |
| Injector heater | Yes | Yes |
| Injector temperature (°C) | 110 | 110 |

According to Jason Dunbar from Varian Canada, the minimum detection limits of this GC for all the above listed gases are approximately 50 ppm (by volume).

4.2.14.1 GC calibration and verification

Based on the gas concentration ranges found in Fedorak *et al.* (2000), the expected concentration ranges for the gases in this project were determined. Four levels of external calibration were used for most of the gases, with the exception of $H_2S$, for which only three levels of external calibration were used. For preparing the desired standard concentrations, pure standard gas, helium gas (as balance gas), a 1-L Tedlar bag, a soap-film flowmeter, and a stopwatch were used.

For $H_2S$, only three level calibrations were used, because of the difficulty in preparing the standard concentrations. The three levels of standard $H_2S$ gas were prepared by Aaron Gatzke in Maxxam Analytics Inc. (Edmonton, Alberta).

The GC calibration used the GC parameters as shown in Table 4-2. The Tedlar bag containing standard concentration gas was directly connected to GC

via a 4-cm long Tygon tubing. At the calibration, the sampling time was set to be 30 s.

After establishing the calibration curves, some verification runs were conducted using individual or mixed standard gases in the Environmental Engineering Laboratory. The verifications used the same method as the calibration. The calibration and verification of this GC are summarised at Tables A-1 and A-2 in Appendix A, respectively.

4.2.14.2 Gas analysis by GC for Tedlar bag gas in Systems 1, 3, and 4

The GC analysis on the Tedlar bag gas in Systems 1, 3, and 4 used the GC parameters in Table 4-2. The Tedlar bag was directly connected to the GC via a 4-cm long Tygon tubing. The sampling time was set to be 30 s. For each Tedlar bag gas, triplicate analysis was conducted.

4.2.14.3 Gas analysis by GC for in-situ column headspace gas in System 2

The GC analysis on the column headspace gas in System 2 used the GC parameters in Table 4-2. The column headspace was directly connected to the GC via a 40-cm long stainless steel tubing with 1.6-mm OD. The sampling time was set to be 60 s. For each headspace gas, triplicate analysis was conducted.

4.2.14.4 Gas analysis by GC for trapped gas in MFT and CT

The GC analysis on the trapped gas in MFT and CT for all systems used the GC parameters in Table 4-2. The serum bottle containing the MFT and CT samples for trapped gas analysis (see Section 4.2.12) was directly connected to the GC via a 10-cm long stainless steel tubing with 1.6-mm OD. The sampling time was set to be 30 s. For each trapped gas sample, triplicate analysis was conducted.

*4.2.15 AVS analysis on MFT and CT samples*

AVS are the sum of amorphous iron monosulphide (FeS), mackinawite ($FeS_{1-x}$), greigite ($Fe_3S_4$) (Duan *et al.* 1997; Morse *et al.* 1987), as well as sulphides of other metals such as ZnS and CdS (Gerard *et al.* 1998; Lasorsa *et al.* 1996). Because AVS species are metastable and reactive, either by oxidation or by subsequent reduction to $FeS_2$ (pyrite), it is reasonable to assume that the presence of AVS is an indicator of recent $SO_4^{2-}$ reduction (Kennedy *et al.* 1999).

AVS are usually determined by first liberating $H_2S$ from the sulphides using cold acid (1 to 6 M HCl), and then distillating and trapping of the released $H_2S$, which can be analysed by several methods. More severe acid treatment, often with heating or with the addition of other chemicals, releases sulphides from other compounds (e.g. pyrite).

In cold acid extraction, the amorphous FeS and mackinawite are quantitatively recovered, but the recovery of greigite is incomplete. Under

harsher conditions, such as hot HCl, or cold 6 M HCl plus $SnCl_2$, greigite and newly formed fine-grained synthetic pyrite are recovered. The presence of $SnCl_2$ in the HCl distillation is believed to help diminish the Fe(III) interference that may oxidize sulphide to elemental sulphur ($S^0$) in acid distillation (Duan *et al.* 1997).

Sampling, handling, and analytical techniques that minimise the effect of oxidation are critical for the accurate measurement of AVS. Sulphide is best maintained when samples are handled under a nitrogen atmosphere, stored at 4°C or frozen at –20°C and analysed within 2 wk after sample collection (Lasorsa and Casas 1996).

Through the discussions with the personnel at a commercial laboratory (ALS Environmental, Vancouver, British Columbia), the adopted AVS analysis method for this project was as follows. The AVS analysis used 6 M cold HCl plus $SnCl_2$ to digest the sample. Thus, it is assumed that the analysed sulphur pool is the AVS plus fine-graded (newly formed) synthetic pyrite. The sampling was completed under a $N_2$ atmosphere. The AVS samples were kept at 4°C in a $N_2$ atmosphere during storage and delivery, and were analysed within 14 d after sample collection.

*4.2.16 Column and sample notations*

Through discussions with Dr. MacKinnon, Syncrude Canada Ltd., the column and sample notations for this project were finalised, as shown in Tables A-3, A-4, and A-5 in Appendix A.

## 4.3 Some definitions and symbols for MFT and CT densifications

For simplifying the presentation and discussion, the following definitions are made and symbols are used throughout this thesis and the Appendices.

*4.3.1 Total volume change (Vt, vol%)*

The total volume is the combined volume of cap water and total tailings (MFT and/or CT) for Systems 1, 3, and 4. The total volume change (Vt) is defined in Equation 4-1.

$$Vt=((H_t-H_0)/H_0)\times 100 \quad \text{.....(4-1)}$$

where $H_0$ is the volume of "MFT and CT", of "MFT', and of "CT" for Systems 1, 3, and 4 at incubation time 0, respectively; $H_t$ is the volume of "cap water and MFT and CT", of "cap water and MFT', and of "cap water and CT" for Systems 1, 3, and 4 at a specific incubation time, respectively. Note, at

incubation time 0, it is assumed there was no cap water in the columns of each system.

Based on the definition, a positive value of Vt indicates an increase in the total volume and a negative value of Vt indicates a decrease in the total volume, relative to the total volume at incubation time 0. The Vt is specifically denoted as Vt(1), Vt(3), Vt(4) (vol%) for Systems 1, 3, and 4, respectively.

*4.3.2 Total tailings volume change (Vtt, vol%)*

The total tailings volume is the volume of "MFT and CT" for Systems 1 and 2; the volume of "MFT" or "CT" for Systems 3 and 4, respectively. The total tailings volume change (Vtt) is defined in Equation 4-2.

$$Vtt=((H_{tt}-H_0)/H_0)\times 100 \quad \text{.....(4-2)}$$

where $H_0$ is the volume of "MFT and CT" for Systems 1 and 2, the volume of "MFT' or "CT" for Systems 3 and 4 at incubation time 0, respectively; $H_{tt}$ is the volume of "MFT and CT" for Systems 1 and 2, the volume of "MFT" or "CT" for Systems 3 and 4 at a specific incubation time, respectively.

Based on the definition, a positive value of Vtt indicates an increase in the total tailings volume and a negative value of Vtt indicates a decrease in the

total tailings volume, relative to the total tailings volume at incubation time 0. The Vtt is specifically denoted as Vtt(1), Vtt(2), Vtt(3), and Vtt(4) (vol%) for Systems 1, 2, 3, and 4, respectively.

*4.3.3 Individual MFT or CT tailings volume change ($V_{MFT}$, $V_{CT}$, vol%)*

The individual MFT or CT tailings volume is the volume of individual "MFT" or "CT" for each system. The individual MFT or CT tailings volume change ($V_{MFT}$ or $V_{CT}$) is defined in Equations 4-3 and 4-4, respectively.

$$V_{MFT}=((H_{MFT}-H_{MFT0})/H_{MFT0})\times 100 \quad \text{.....(4-3)}$$

$$V_{CT}=((H_{CT}-H_{CT0})/H_{CT0})\times 100 \quad \text{.....(4-4)}$$

where $H_{MFT0}$ and $H_{CT0}$ are the volumes of "MFT" or "CT" for Systems 1, 2, 3, and 4 at incubation time 0; $H_{MFT}$ and $H_{CT}$ are the volumes of "MFT" or "CT" for Systems 1, 2, 3, and 4 at a specific incubation time.

Based on the definition, a positive value of $V_{MFT}$ or $V_{CT}$ indicates an increase in the individual MFT or CT tailings volume and a negative value of $V_{MFT}$ or $V_{CT}$ indicates a decrease in the individual MFT or CT tailings volume, relative to the individual MFT or CT tailings volume at incubation time 0. The $V_{MFT}$ or $V_{CT}$ is specifically denoted as $V_{MFT(1)}$, $V_{CT(1)}$, $V_{MFT(2)}$, $V_{CT(2)}$, $V_{MFT(3)}$, and $V_{CT(4)}$ (vol%) for Systems 1, 2, 3, and 4, respectively.

It should be noted that by definition:

$V_{MFT(3)}$=Vtt(3);

$V_{CT(4)}$=Vtt(4).

*4.3.4 Cap water volume (Vcw, vol%)*

The cap water volume (Vcw) is defined in Equation 4-5.

$$Vcw=(H_{cw}/H_0)\times 100 \quad (4\text{-}5)$$

where $H_0$ is the volume of "MFT and CT", of "MFT", and of "CT" for Systems 1, 3, and 4 at incubation time 0, respectively; $H_{cw}$ is the volume of cap water for Systems 1, 3, and 4 at a specific incubation time, respectively.

The Vcw is specifically denoted as Vcw(1), Vcw(3), and Vcw(4) (vol%) for Systems 1, 3, and 4, respectively.

*4.3.5 Cap water producing rate (Rcw, mL/d)*

The cap water producing rate (Rcw) is defined in Equation 4-6.

$$Rcw = (H_{cw2}-H_{cw1})/(t_2-t_1) \quad (4\text{-}6)$$

where $t_1$ and $t_2$ are any two consecutive sampling times with $t_2 > t_1$; $H_{cw1}$ and $H_{cw2}$ are the accumulated volumes of cap water for Systems 1, 3, and 4 at the sampling times of $t_1$ and $t_2$, respectively.

Based on the definition, Rcw is the average cap water producing rate between any two consecutive sampling times. The Rcw is specifically denoted as Rcw(1), Rcw(3), and Rcw(4) (mL/d) for Systems 1, 3, and 4, respectively.

*4.3.6 Headspace volume (Vhs, mL)*

The headspace volume (Vhs) is the headspace volume of those sacrificed columns in Systems 1, 3, and 4. The Vhs is specifically denoted as Vhs(1), Vhs(3), and Vhs(4) (mL) for Systems 1, 3, and 4, respectively.

## 4.4 Column setup

*4.4.1 Column setup in Systems 1, 3, and 4.*

The MFT and CT placements for columns in Systems 1, 3, and 4 were conducted on November 4, 5, and 6, 2002. The procedures for column filling were discussed previously (Section 4.2.4).

The MFT and CT were stored in the dark storage room at 4°C before use. Because the CT had been in storage for quite a long time, the chemical and microbiological processes may have proceeded to some degree and lead to

lowered $SO_4^{2-}$ and $Ca^{2+}$ concentrations in the CT porewater. According to the suggestion from Dr. MacKinnon, Syncrude Canada Ltd., the CT was amended with $Na_2SO_4$ and $CaSO_4 \bullet 2H_2O$ salts at a suitable dosage to raise the $SO_4^{2-}$ and $Ca^{2+}$ levels comparable to those in the fresh CT.

A tracer ion, $Br^-$, was added to the CT in the form of NaBr. Because of its low degree of interactions with other species in the treat materials, $Br^-$ was used as a tracer to follow the transit of the CT porewater release through the MFT layer in Systems 1 and 2. As the tracer in CT and MFT matrices, $Br^-$ was selected over $F^-$ after some preliminary tests.

No chemical amendments were applied to the MFT.

The chemical additions in CT were conducted at the time of column filling. The chemicals were well mixed with the CT. Table 4-3 lists the planned chemical dosages in the CT. The actual chemical additions might be slightly different from the planned dosages due to the errors in weighing the chemical dosages and measuring the CT volumes.

**Table 4-3. The planned chemical dosages in CT for Systems 1 and 4.**

| Chemical | Planned dosage (g/L of CT) |
|---|---|
| NaBr | 0.22 |
| $Na_2SO_4$ | 0.56 |
| $CaSO_4 \bullet 2H_2O$ | 0.06 |

Prior to column filling, CT mixing was conducted in a 100-L plastic container in the air and the MFT mixing was conducted in a 200-L plastic container under $N_2$ atmosphere. The CT and MFT materials for filling in

Systems 1, 3, and 4 were divided into several mixings. The baseline samples of all different mixings of MFT and CT were taken and analysed individually. Table 4-4 summarises the MFT and CT mixings and baseline sample notations for Systems 1, 3, and 4.

**Table 4-4. MFT and CT mixings and baseline sample notations for Systems 1, 3, and 4.**

| Materials | Volume of each mixing (L) | Date of mixing and filling | Columns filled with | Baseline sample notation |
|---|---|---|---|---|
| MFT | 135 | 4 Nov.-02 | C1-1, ..., C1-20. | Base MFT 1 |
| | 135 | 6 Nov.-02 | C3-1, ..., C3-12. | Base MFT 2 |
| CT | 70 | 5 Nov.-02 | C1-1, ..., C1-11 | Base CT 1 |
| | 70 | 5 Nov.-02 | C1-12, ..., C1-20 | Base CT 2 |
| | 80 | 6 Nov.-02 | C4-1, ..., C4-12 | Base CT 3 |

The column filling and incubation starting times for Systems 1, 3, and 4 are summarised in Table A-6 in Appendix A. Figure 4-4 shows the columns of Systems 1, 3, and 4 in the incubation room.

**Figure 4-4. The columns of Systems 1, 3, and 4 in the incubation room.**

*4.4.2 Column setup in System 2*

The MFT and CT placements for columns in System 2 were conducted on December 9 and 10, 2002. The procedures for column filling in System 2 were discussed previously in Section 4.2.4.

Chemical amendments and $Br^-$ tracer addition in the form of NaBr in CT for System 2 were conducted. Table 4-5 shows the planned chemical dosages in the CT of System 2. The actual chemical additions might be slightly different from the planned dosages due to the errors in weighing the chemical dosages

and measuring the CT volumes. The six columns in System 2 were divided into three groups in terms of chemical dosage in CT.

**Table 4-5. The planned chemical dosages in CT of System 2.**

| Column | Planned dosage (g/L of CT) | | | |
|---|---|---|---|---|
| | NaBr | $Na_2SO_4$ | $CaSO_4 \cdot 2H_2O$ | $CH_3COONa \cdot 3H_2O$ |
| C2-1, C2-2, and C2-3 | 0.22 | 0.56 | 0.06 | 0 |
| C2-4 and C2-5 | 0.22 | 1.11 | 0.22 | 0 |
| C2-6 | 0.22 | 1.11 | 0.22 | 1.00 |

As shown in Table 4-5, The CT in columns C2-1, C2-2, and C2-3 had the same chemical amendments ($Na_2SO_4$, and $CaSO_4 \cdot 2H_2O$) as in System 1. The CT in columns C2-4 and C2-5 had higher chemical amendments of $Na_2SO_4$ and $CaSO_4 \cdot 2H_2O$ than in System 1. In column C2-6, the CT had the same dosages of $Na_2SO_4$ and $CaSO_4 \cdot 2H_2O$ amendments as in columns of C2-4 and C2-5, but with the addition of sodium acetate ($CH_3COONa \cdot 3H_2O$). The purpose of adding this $CH_3COONa \cdot 3H_2O$ was to see if it would stimulate the microbial (methanogens and SRB) activity and hence causing related physical and chemical changes in the MFT and CT.

Prior to column filling, the MFT and CT for System 2 were also well mixed in several mixings. The chemical amendments were applied to CT at CT mixing. The baseline samples of all different mixings of MFT and CT were taken and analysed individually. Table 4-6 summarises the MFT and CT mixings and baseline sample notations for System 2.

**Table 4-6. MFT and CT mixings and baseline sample notations for System 2.**

| Materials | Volume of each mixing (L) | Date of mixing and filling | Columns filled with | Baseline sample notation |
|---|---|---|---|---|
| MFT | 25 | 9 Dec.-02 | C2-1, ..., C2-6 | Base MFT 3 |
| CT | 25 | 10 Dec.-02 | C2-1, C2-2, C2-3 | Base CT 4 |
| | 25 | 10 Dec.-02 | C2-4, C2-5 | Base CT 5 |
| | 12 | 10 Dec.-02 | C2-6 | Base CT 6 |

The column filling and incubation starting times for System 2 are summarised in Table A-7 in Appendix A. Figure 4-5 shows the columns of System 2 in the incubation room.

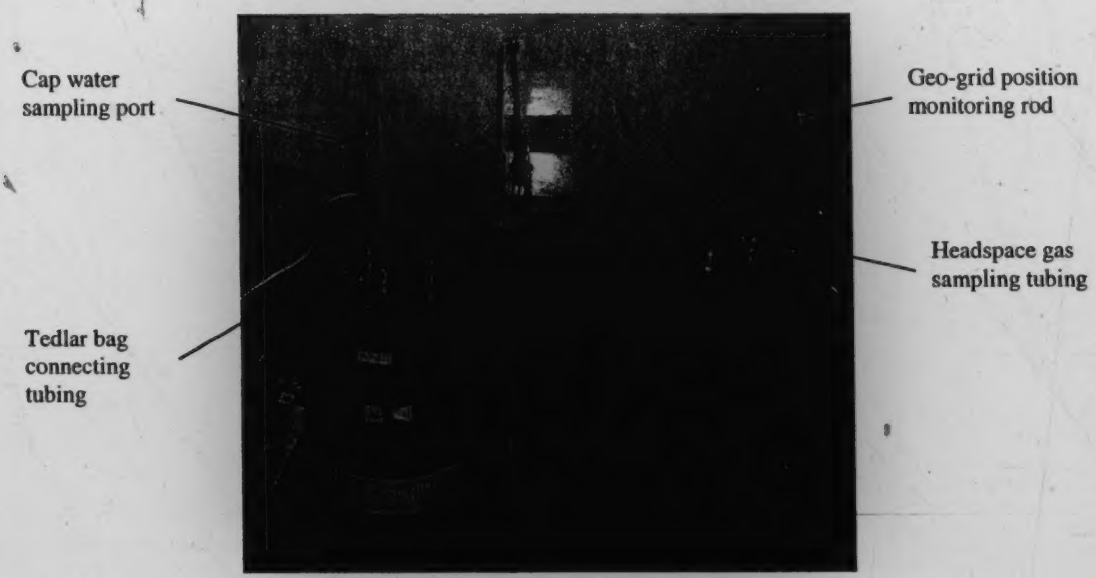



**Figure 4-5. The columns of System 2 in the incubation room.**

## 5.0 Results

Columns in Systems 1, 3, and 4 were sacrificially sampled on a regular basis. Columns in System 2 were sacrificed only at the end of 1 y of incubation. The incubation time and sacrificed columns for Systems 1, 2, 3, and 4 are summarised in Table 5-1. As an example for Table 5-1, 3 (C1-2) means the column C1-2 was sacrificed after 3 d of incubation.

**Table 5-1. Incubation time and sacrificed columns for Systems 1, 2, 3, and 4.**

| System | $t_{incu}$ (d) and sacrificed columns | Number of sacrificed columns |
|---|---|---|
| 1 (Cap water/MFT/CT) | 3 (C1-2), 15 (C1-3), 29 (C1-4), 62 (C1-5), 91 (C1-6), 121 (C1-12), 153 (C1-8), 182 (C1-9), 213 (C1-10), 244 (C1-11), 267 (C1-13), 303 (C1-14), 335 (C1-15), 366 (C1-16). | 14 |
| 2 (Cap water/MFT/CT) | 352 (C2-1, C2-4, C2-6) | 3 |
| 3 (Cap water/MFT) | 28 (C3-1), 61 (C3-2), 90 (C3-3), 120 (C3-4), 152 (C3-5), 181 (C3-6), 212 (C3-7), 243 (C3-8), 266 (C3-9), 302 (C3-10), 334 (C3-11), 365 (C3-12) | 12 |
| 4 (Cap water/CT) | 28 (C4-1), 61 (C4-2), 90 (C4-3), 120 (C4-4), 152 (C4-5), 181 (C4-6), 212 (C4-7), 243 (C4-8), 266 (C4-9), 302 (C4-10), 334 (C4-11), 365 (C4-12) | 12 |

1). $t_{incu}$=incubation time.

The regular monitorings during incubation and analyses at sacrificial sampling for Systems 1, 2, 3, and 4 were given in Section 4.2.

## 5.1 MFT and CT densifications and water release

### *5.1.1 MFT densification and water release in System 3 (Cap water/MFT)*

Figures B-1, B-2, B-3, and B-4 in Appendix B summarise the Vt(3), Vtt(3), Vcw(3), and Rcw(3) values over 1 y of incubation for the columns in System 3. Table 5-2 summarises the relevant parameters for the columns in System 3 at the end of specific incubation periods.

**Table 5-2. Summary of relevant parameters at the end of specific incubation period in System 3.**

| Column | $t_{incu}$ (d) | Vt(3) (vol%) | Vtt(3) (vol%) | Vcw(3) (vol%) | Rcw(3) (mL/d) | Vhs(3) (mL) |
|---|---|---|---|---|---|---|
| C3-1 | 28 | 4.4 | 1.3 | 3.1 | 7.5 | 3970 |
| C3-2 | 61 | 10.6 | 2.7 | 7.9 | 6.6 | 3560 |
| C3-3 | 90 | 13.0 | 3.7 | 9.3 | 0.9 | 3450 |
| C3-4 | 120 | 9.3 | -2.0 | 11.3 | 4.1 | 3690 |
| C3-5 | 152 | 10.2 | -3.3 | 13.5 | 3.2 | 3660 |
| C3-6 | 181 | 10.1 | -3.7 | 13.7 | 1.9 | 3650 |
| C3-7 | 212 | 8.6 | -5.1 | 13.8 | 1.1 | 3730 |
| C3-8 | 243 | 8.6 | -5.7 | 14.3 | -0.7 | 3710 |
| C3-9 | 266 | 8.1 | -7.1 | 15.2 | 0.5 | 3740 |
| C3-10 | 302 | 7.5 | -8.0 | 15.5 | 0.9 | 3730 |
| C3-11 | 334 | 7.9 | -8.1 | 15.9 | 1.4 | 3730 |
| C3-12 | 365 | 6.9 | -8.8 | 15.7 | 0.8 | 3810 |

Table B-1 in Appendix B lists the absolute heights of cap water and MFT at time 0 and at column sacrificing for each sacrificed column in System 3. It is noted that the height of cap water at time 0 is zero. Based on Table B-1, Figure 5-1 shows the relative heights of cap water and MFT in the sacrificed columns of System 3 over 1 y of incubation.

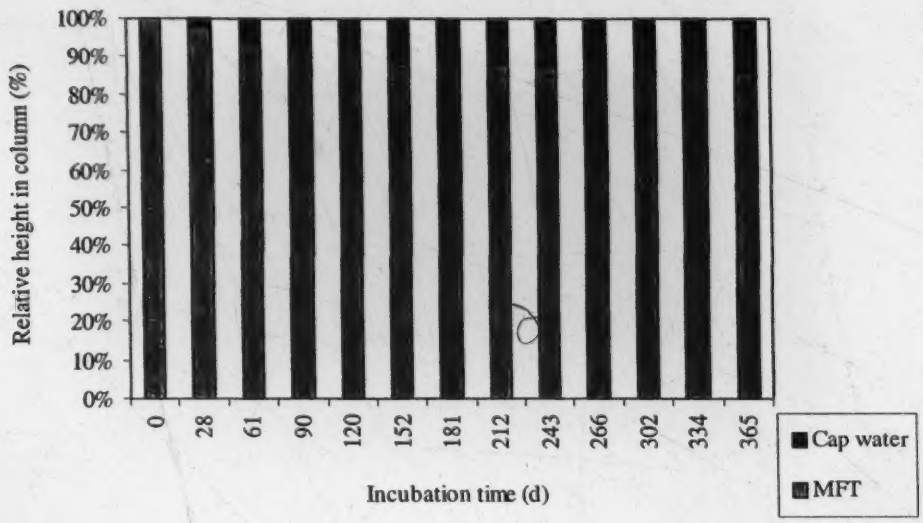



**Figure 5-1. Relative heights of cap water and MFT in System 3.**

*5.1.2 CT densification and water release in System 4 (Cap water/CT)*

Figures B-5, B-6, B-7, and B-8 in Appendix B summarise the Vt(4), Vtt(4), Vcw(4), and Rcw(4) values over 1 y of incubation for the columns in System 4. Table 5-3 summarises the relevant parameters for the columns in System 4 at the end of specific incubation periods.

**Table 5-3. Summary of relevant parameters at the end of specific incubation period in System 4.**

| Column | $t_{incu}$ (d) | Vt(4) (vol%) | Vtt(4) (vol%) | Vcw(4) (vol%) | Rcw(4) (mL/d) | Vhs(4) (mL) |
|---|---|---|---|---|---|---|
| C4-1 | 28 | 0.0 | -22.5 | 22.5 | 15.1 | 4260 |
| C4-2 | 61 | -0.4 | -28.2 | 27.9 | 6.6 | 4300 |
| C4-3 | 90 | 0.0 | -30.5 | 30.5 | 2.6 | 4270 |
| C4-4 | 120 | 0.0 | -31.0 | 31.0 | 1.1 | 4270 |
| C4-5 | 152 | 0.0 | -31.4 | 31.4 | 1.1 | 4220 |
| C4-6 | 181 | 0.0 | -31.8 | 31.8 | 0.8 | 4280 |
| C4-7 | 212 | 0.0 | -31.9 | 31.9 | 0.4 | 4350 |
| C4-8 | 243 | -0.4 | -31.8 | 31.4 | 0.0 | 4310 |
| C4-9 | 266 | -0.7 | -32.6 | 31.9 | 0.0 | 4340 |
| C4-10 | 302 | -0.4 | -32.1 | 31.7 | 0.3 | 4270 |
| C4-11 | 334 | -0.7 | -32.6 | 31.9 | 0.0 | 4410 |
| C4-12 | 365 | -0.4 | -31.9 | 31.5 | 0.0 | 4360 |

Table B-2 in Appendix B lists the absolute heights of cap water and CT at time 0 and at column sacrificing for each sacrificed column in System 4. It is noted that the height of cap water at time 0 is zero. Based on Table B-2, Figure 5-2 shows the relative heights of cap water and CT in the sacrificed columns of System 4 over 1 y of incubation.

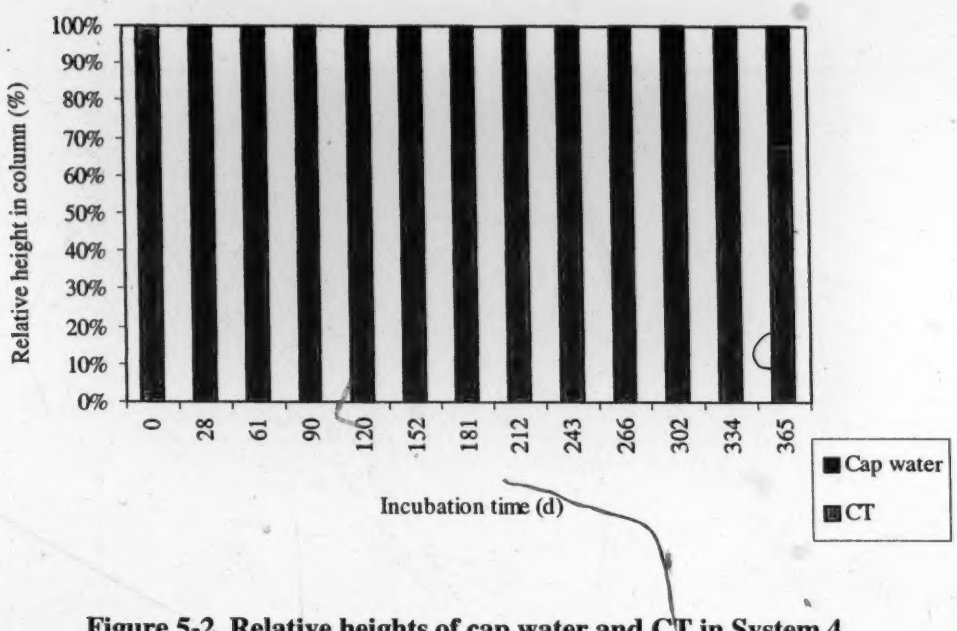



**Figure 5-2. Relative heights of cap water and CT in System 4.**

*5.1.3 MFT and CT densifications and water release in System 1 (Cap water/MFT/CT)*

Figures B-9, B-10, B-11, B-12 in Appendix B summarise the Vt(1), Vtt(1), Vcw(1), and Rcw(1) values over 1 y of incubation for the columns in System 1. Table 5-4 summarises the relevant parameters for the columns in System 1 at the end of specific incubation periods.

**Table 5-4. Summary of relevant parameters at the end of specific incubation period in System 1.**

| Column | $t_{incu}$ (d) | Vt(1) (vol%) | Vtt(1) (vol%) | Vcw(1) (vol%) | Rcw(1) (mL/d) | $V_{CT(1)}$ (vol%) | $V_{MFT(1)}$ (vol%) | Vhs(1) (mL) |
|---|---|---|---|---|---|---|---|---|
| C1-2 | 3 | -0.1 | -4.8 | 4.6 | 90.4 | -7.3 | 0.4 | 1130 |
| C1-3 | 15 | -1.4 | -11.8 | 10.5 | 28.3 | -19.3 | 1.5 | 1330 |
| C1-4 | 29 | -0.6 | -15.5 | 14.8 | 18.8 | -23.4 | 0.0 | 1220 |
| C1-5 | 62 | 0.2 | -18.3 | 18.6 | 8.5 | -29.7 | 4.8 | 1160 |
| C1-6 | 91 | -0.6 | -20.1 | 19.5 | 5.2 | -29.7 | -1.8 | 1210 |
| C1-12 | 121 | 0.7 | -21.2 | 22.0 | 3.0 | -30.7 | -2.2 | 1100 |
| C1-8 | 153 | -2.8 | -22.4 | 19.5 | 0.7 | -33.1 | -1.1 | 1410 |
| C1-9 | 182 | -3.4 | -23.4 | 20.0 | 0.4 | -32.6 | -4.8 | 1450 |
| C1-10 | 213 | 1.2 | -20.3 | 21.6 | 0.7 | -32.1 | 3.3 | 1040 |
| C1-11 | 244 | 1.6 | -20.0 | 21.6 | 0.4 | -30.0 | -0.4 | 995 |
| C1-13 | 267 | 1.8 | -22.0 | 23.8 | -1.0 | -32.6 | -0.4 | 950 |
| C1-14 | 303 | 1.7 | -23.3 | 25.0 | 0.6 | NA | NA | 1010 |
| C1-15 | 335 | -2.1 | -24.8 | 22.8 | 0.4 | -34.9 | -5.1 | 1310 |
| C1-16 | 366 | 2.8 | -22.1 | 24.9 | 1.5 | -33.8 | 1.1 | 893 |
| C1-1 | 366 | -8.8 | -26.2 | 17.4 | 0.0 | NA | NA | NA |
| C1-17 | 366 | -2.7 | -24.6 | 21.9 | 1.5 | NA | NA | NA |
| C1-18 | 366 | 2.3 | -22.4 | 24.7 | 1.1 | NA | NA | NA |
| C1-19 | 366 | -3.4 | -25.2 | 21.8 | 0.0 | NA | NA | NA |
| C1-20 | 366 | -6.4 | -26.1 | 19.7 | 0.0 | NA | NA | NA |

1). NA=not available.

Table B-3 in Appendix B lists the absolute heights of cap water, MFT, and CT at time 0 and at column sacrificing for each sacrificed column in System 1. The separated MFT and CT heights of those sacrificed columns in System 1 are determined by measuring the position of Geo-grid at column sacrificing. It is noted that the height of cap water at time 0 is zero. Based on Table B-3, Figure 5-3 shows the relative heights of cap water, MFT, and CT in the sacrificed columns of System 1 over 1 y of incubation.

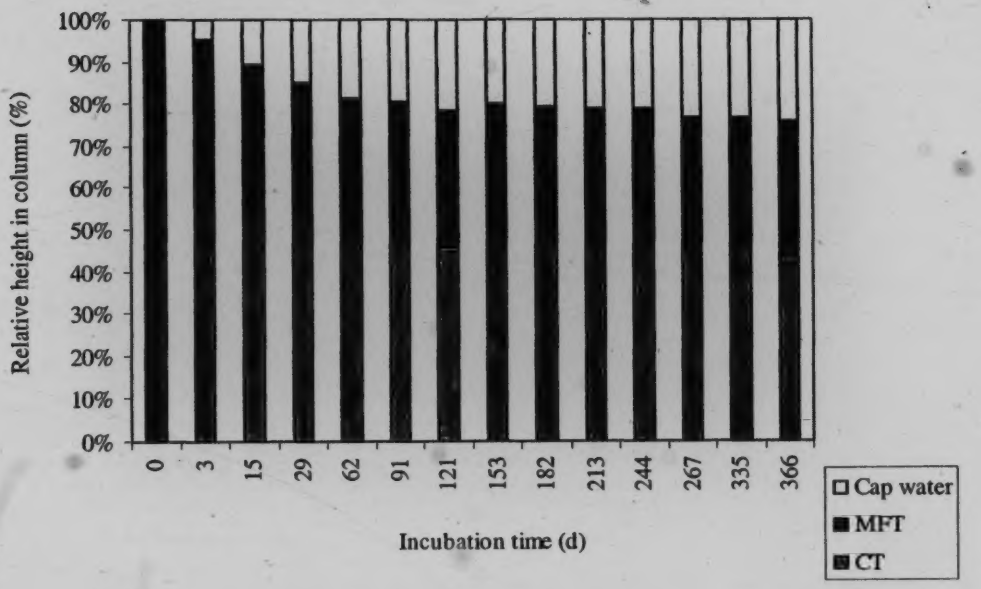



**Figure 5-3. Relative heights of cap water, MFT, and CT in System 1.**

*5.1.4 MFT and CT densifications and water release in System 2 (Cap water/MFT/CT)*

Figures B-13, B-15, and B-14 in Appendix B summarise the Vtt(2), $V_{CT(2)}$, and $V_{MFT(2)}$ values over 1 y of incubation for the columns in System 2. Table 5-5 summarises the relevant parameters for the columns in System 2 at the end of 1 y of incubation.

**Table 5-5. Summary of relevant parameters at the end of 1 y of incubation in System 2.**

| Column | $t_{incu}$ (d) | Vtt(2) (vol%) | $V_{CT(2)}$ (vol%) | $V_{MFT(2)}$ (vol%) |
|---|---|---|---|---|
| C2-1 | 352 | -26.2 | -31.8 | -14.9 |
| C2-2 | 352 | -22.5 | -32.8 | -1.8 |
| C2-3 | 352 | -26.0 | -34.4 | -9.2 |
| C2-4 | 352 | -27.7 | -36.9 | -9.6 |
| C2-5 | 352 | -27.7 | -37.8 | -7.7 |
| C2-6 | 352 | -26.8 | -37.4 | -6.2 |

## 5.2 Headspace gas analysis

### *5.2.1 Tedlar bag gas volume and GC analysis in Systems 1, 3, and 4*

Table B-4 in Appendix B summarises the Tedlar bag gas volume and GC analysis results for the major sacrificed columns in Systems 1, 3, and 4.

### *5.2.2 In-situ headspace gas GC analysis in System 2*

Table B-5 in Appendix B summarises the frequent in-situ headspace gas analysis results for the columns in System 2, over 1 y of incubation.

## 5.3 Physical and chemical analyses

The physical and chemical analyses include pH, EC, cations ($Ca^{2+}$, $Mg^{2+}$, $Na^{+}$, $K^{+}$, $NH_4^{+}$), anions ($SO_4^{2-}$, $S^{2-}$, $PO_4^{3-}$, $NO_3^{-}$, $NO_2^{-}$, $Cl^{-}$), trace elements (Al, Fe), AVS, Eh, solids content, residual bitumen, particle size distribution, and $S^{2-}$. The $Br^{-}$, $F^{-}$, and $SO_4^{2-}$ ions, and Al and Fe trace metals in some samples were non-detectable or below detection limits (BDL). The detection limits are 0.5

mg/L for $Br^-$, $F^-$, and $SO_4^{2-}$, and 0.01 mg/L for Al and Fe (Dr. MacKinnon, Syncrude Canada Ltd., personal communication).

### *5.3.1 Physical and chemical analyses on baseline MFT and CT*

Table B-6 in Appendix B summarises the results of physical and chemical analyses on the baseline MFT and CT samples for Systems 1, 2, 3, and 4. The baseline MFT and CT samples were taken at the column fillings, and their data serve as the time 0 baseline data.

### *5.3.2 Physical and chemical analyses in System 3 (Cap water/MFT)*

Table B-7 in Appendix B summarises the results of physical and chemical analyses on the cap water, MFT porewater, and MFT samples for all the sacrificed columns in System 3 over 1 y of incubation.

### *5.3.3 Physical and chemical analyses in System 4 (Cap water/CT)*

Table B-8 in Appendix B summarises the results of physical and chemical analyses on the cap water, CT porewater, and CT samples for all the sacrificed columns in System 4 over 1 y of incubation.

*5.3.4 Physical and chemical analyses in System 1 (Cap water/MFT/CT)*

Table B-9 in Appendix B summarises the results of physical and chemical analyses on the cap water, MFT and CT porewaters, and MFT and CT samples for all the sacrificed columns in System 1 over 1 y of incubation.

*5.3.5 Physical and chemical analyses in System 2 (Cap water/MFT/CT)*

Table B-10 in Appendix B summarises the results of physical and chemical analyses on the cap water, MFT and CT porewaters, and MFT and CT samples for the columns in System 2 over 1 y of incubation.

## 5.4 Trapped gas analysis

*5.4.1 Trapped gas analysis in baseline MFT and CT*

Table B-11 in Appendix B summarises the results of trapped gas GC analysis on all baseline MFT and CT samples for Systems 1, 2, 3, and 4.

*5.4.2 Trapped gas analysis in Systems 1, 2, 3, and 4*

Table B-12 in Appendix B summarises the results of trapped gas GC analysis on the MFT and CT samples in major sacrificed columns of Systems 1, 2, 3, and 4.

## 5.5 MPN analysis in System 1

Table B-13 in Appendix B summarises the MPN analysis results on the MFT and CT samples for specific sacrificed columns in System 1.

## 6.0 Discussion

Systems 3 and 4 contained only MFT or CT, respectively, and served as controls for the main systems. Systems 1 and 2 contained a combination of overlying MFT and underlying CT, and were the main systems to be studied. System 2 was designed in particular to allow for regular in-situ cap water and headspace gas analyses, and Geo-grid position monitoring without sacrificial sampling.

### 6.1 MFT and CT densifications and water release

Figures 5-1, 5-2, and 5-3 visually shows the changes, over incubation time, of relative heights of cap water, MFT, and CT layers in the sacrificed columns of Systems 3, 4, and 1, respectively. The detailed discussions on the MFT and CT densifications and water release are presented as follows.

#### *6.1.1 Total volume change (Vt) in Systems 3, 4, and 1*

For System 3 (Cap water/MFT), Figure B-1 shows that over 1 y of incubation, the Vt(3) values among the columns were quite similar. The Vt(3) increased quickly from the beginning of the incubation and reached a maximum value in the range of 12.3 to 13.5 vol% in 90 d of incubation among the columns. The Vt(3) then decreased with time and reached 6.9 vol% at 365 d of incubation.

A similar total volume increase in Syncrude MFT in the early incubation was also noted by Fedorak *et al.* (2000).

Fedorak *et al.* (2000) proposed the production of biogenic gas as the possible cause for the total volume increase in MFT deposit, and had observed evident bubbles entrapped in MFT. The high volume of Tedlar bag gas accumulated in System 3 (see Table B-4) indicated that active gas production occurred in the MFT of System 3. Visual observations also showed obvious gas bubbles that rose to the surface of the cap water in columns of System 3.

For System 4 (Cap water/CT), Figure B-5 shows that over 1 y of incubation, the Vt(4) values among the columns were quite small and within 0 to -0.7 vol% (see Figure B-5). These small Vt(4) values during incubation may be due to the small evolved and entrapped gases and the uniform settling and densification in CT. The Tedlar bag gas volume measurement showed little gas production in System 4 (see Table B-4), and no gas bubbles were observed on the cap water surface of columns in System 4.

For System 1 (Cap water/MFT/CT), Figure B-9 shows that after 1 y of incubation, the Vt(1) values among the columns were in the range of -8.8 to 2.8 vol%, indicating a large variation of Vt(1) among different columns.

Possible explanations for the decrease in total volume are the densification of MFT and/or CT, and the release of entrapped gases from the MFT and/or CT layers. Densifications of MFT and/or CT will increase the tailings density and reduce the tailings volume. Gas entrapping into the MFT

and/or CT at column filling is possible. The release of the entrapped gas during incubation will reduce the tailings volume and thus the total volume.

One possible explanation for the increase in the total volume is the trapping and accumulation of newly evolved biogenic gases in the MFT and/or CT layers during incubation.

System 1 showed a large variation in Vt value among the columns over 1 y of incubation. Some columns have positive Vt values while others have negative Vt values over time (see Figure B-9). It is possible that this is in part due to the heterogeneity of MFT and CT materials in different columns. The heterogeneity of MFT and CT materials will lead to different solids densification, gas production and trapping.

### *6.1.2 Total and individual tailings volume changes (Vtt, $V_{MFT}$, and $V_{CT}$) in Systems 3, 4, 1, and 2*

The total tailings volume change (Vtt) or individual tailings volume change ($V_{MFT}$ and $V_{CT}$) can indicate the expansion or shrinkage of the volumes of total "MFT + CT" or individual MFT and CT tailings.

#### 6.1.2.1 Total tailings volume change of MFT (Vtt(3)) in System 3 (Cap water/MFT)

Figure B-2 shows that the MFT tailings volume expanded steadily in the first 60 to 90 d of incubation and reached maximum values of Vtt(3) between 3.6 and 4.6 vol% among the columns of System 3 at 77 d of incubation. After

this time, the Vtt(3) dropped steadily and reached 0 vol% at about 110 d of incubation, and -8.8 vol% at 365 d of incubation.

Table 6-1 lists some Vtt(3) data among the columns of System 3 at specific incubation times, together with solids contents of MFT for the sacrificed columns in System 3 (from Table B-7).

**Table 6-1. Tailings volume change and solids content of MFT in System 3 at specific incubation times.**

| $t_{incu}$ (d) | Tailings volume change (vol%) | Solids content (wt%) | |
|---|---|---|---|
| | Vtt(3) | MFT-U | MFT-L |
| 0 | 0 | 38.0 | 38.0 |
| 61 | 2.7 to 3.3 | 39.7 | 40.3 |
| 120 | -1.6 to -2.4 | 41.8 | 41.9 |
| 181 | -3.3 to -5.1 | 42.4 | 43.2 |
| 266 | -6.4 to -7.1 | 46.8 | 43.0 |
| 334 | -8.0 to -8.1 | 45.7 | 46.2 |
| 365 | -8.8 | 44.3 | 45.6 |

1). NA=not available.
2). Incubation time 0 means baseline samples.

Both the Vtt(3) and solids content values showed densification trend of MFT over 1 y of incubation. However, during the early incubation times, Vtt(3) showed an expansion of MFT while the solids content still showed densification of MFT. The trapped gases in the MFT may be a factor to cause this difference. Gases present in the MFT will increase Vtt of MFT but will not affect the solids content results.

6.1.2.2 Total tailings volume change of CT (Vtt(4)) in System 4 (Cap water/CT)

Figure B-6 shows that over 1 y of incubation, the Vtt(4) decreased substantially in the columns of System 4. The most rapid volume reduction of CT tailings in System 4 occurred in the first 60 d of incubation. Among the columns of System 4, the CT volume reduction reached the Vtt(4) values between -27.7 and -28.6 vol% at 61 d of incubation, and -31.9 vol% at 365 d of incubation.

Table 6-2 lists some Vtt(4) data among the columns of System 4 at specific incubation times, together with the solids contents of CT for the sacrificed columns (from Table B-8).

**Table 6-2. Tailings volume change and solids content of CT in System 4 at specific incubation times.**

| $t_{incu}$ (d) | Tailings volume change (vol%) | Solids content (wt%) | |
|---|---|---|---|
| | Vtt(4) | CT-U | CT-L |
| 0 | 0 | 56.6 | 56.6 |
| 61 | -27.7 to -28.6 | 69.3 | 70.9 |
| 120 | -30.6 to –31.3 | 71.3 | 73.3 |
| 181 | -31.8 to -32.4 | 70.4 | 72.8 |
| 212 | -31.4 to -32.6 | 75.7 | 74.1 |
| 266 | -31.7 to -32.6 | 71.5 | 73.2 |
| 334 | -31.9 to -32.6 | 74.9 | 75.9 |
| 356 | -31.9 | 75.9 | 76.4 |

1). NA=not available.
2). Incubation time 0 means baseline samples.

It can be seen from Table 6-2 that the solids content in the sacrificed columns of System 4 increased over time, showing densification of CT over time. This is consistent with the Vtt(4) curve (see Figure B-6). It should be noted

that for both the solids content increase and tailings volume decrease, most of the change occurred in the first 60 d of incubation.

6.1.2.3 Total and individual tailings volume changes (Vtt(1), $V_{MFT(1)}$, $V_{CT(1)}$) in System 1 (Cap water/MFT/CT)

Figure B-10 shows that over 1 y of incubation, the Vtt(1) value among the columns of System 1 decreased steadily, with the most rapid decrease occurring in the first 60 d of incubation. The Vtt(1) values among the columns of System 1 were between –17.0 and -21.2 vol% at 62 d of incubation and between -22.4 and -26.2 vol% at 366 d of incubation (see Figure B-10).

Figure C-1 in Appendix C displays the values of Vtt(1), $V_{MFT(1)}$, and $V_{CT(1)}$ for those sacrificed columns in System 1 over 1 y of incubation. The $V_{MFT(1)}$ and $V_{CT(1)}$ were calculated by measuring the position of Geo-grid at the time of sacrificial sampling.

Figure C-1 shows that the MFT layer expanded slightly in the first 60 d of incubation, reaching a maximum $V_{MFT(1)}$ of 4.8 vol% at 62 d of incubation, and started shrinking thereafter. The MFT volume change reached a $V_{MFT(1)}$ of -4.8 vol% at 182 d of incubation. After this time, the $V_{MFT(1)}$ curve was fluctuating and showed no certain decreasing or increasing trend with time. One likely source of this fluctuation in $V_{MFT(1)}$ is the errors introduced in the measurement of Geo-grid position when sacrificing the columns. Because MFT settles more slowly than CT, the errors in measuring the Geo-grid position will have a greater effect on the accuracy of $V_{MFT(1)}$ data than on that of $V_{CT(1)}$ data.

Most of the CT volume decrease also occurred in the first 60 d of incubation, with $V_{CT(1)}$ at –30.0 vol% at 62 d of incubation and at -33.8 vol% at 366 d of incubation. The decrease in Vtt(1) was mainly caused by the tailings volume decrease in CT layer.

Table 6-3 lists some data of Vtt(1), $V_{MFT(1)}$, and $V_{CT(1)}$ in System 1 at specific incubation times, together with the solids contents of MFT and CT for the sacrificed columns. It should be noted that the Vtt(1) data were derived from all the columns under incubation, while the $V_{MFT(1)}$ and $V_{CT(1)}$ data were derived only from the sacrificed columns.

**Table 6-3. Total and individual tailings volume changes and solids content at specific incubation times in System 1.**

| $t_{incu}$ (d) | Total and individual tailings volume changes (vol%) | | | Solids content (wt%) | | | |
|---|---|---|---|---|---|---|---|
| | Vtt(1) | $V_{MFT(1)}$ | $V_{CT(1)}$ | MFT-U | MFT-L | CT-U | CT-L |
| 0 | 0 | 0 | 0 | 37.9 | 37.9 | 57.2 | 57.2 |
| 62 | -17.0 to -21.2 | 4.8 | -29.7 | 38.3 | 38.8 | 71.4 | 69.2 |
| 121 | -19.2 to -23.8 | -2.2 | -30.7 | 38.9 | 38.8 | 73.3 | 70.9 |
| 182 | -19.4 to -24.0 | -4.8 | -32.6 | 40.1 | 41.3 | 73.1 | 73.8 |
| 267 | -21.7 to -25.4 | -0.4 | -32.6 | 40.0 | 40.4 | 71.0 | 72.8 |
| 335 | -21.7 to -25.9 | -5.1 | -34.9 | 43.0 | 43.9 | 75.6 | 76.3 |
| 366 | -22.4 to -26.2 | 1.1 | -33.8 | 42.5 | 42.9 | 73.2 | 73.5 |

1). Incubation time 0 means baseline samples.
2). The baseline solids content of MFT was 37.9 wt% for all samples in System 1(from 'Base MFT 1').
3). The baseline solids content of CT at 62 and 182 d of incubation was 57.2 wt% (from 'Base CT 1'), and at 121, 267, 335, and 366 d of incubation was 56.5 wt% (from 'Base CT 2').
4). NA=not available.

As shown in Table 6-3, the solids content of CT increased steadily over time and most rapidly in the first 62 d of incubation. This was well matched with the $V_{CT(1)}$ curve. The solids content of MFT also increased steadily over 1 y of incubation. This is not consistent with the fluctuating $V_{MFT(1)}$ data over time. As

discussed above, the fluctuation in $V_{MFT(1)}$ is likely caused by the errors introduced in the measurement of Geo-grid position when sacrificing the columns. The solids content data show that both MFT and CT in System 1 densified to a large extent over time.

6.1.2.4 Total and individual tailings volume changes (Vtt(2), $V_{MFT(2)}$, $V_{CT(2)}$) in System 2 (Cap water/MFT/CT)

Figure B-13 in Appendix B shows that during 352 d of incubation, the Vtt(2) values among the columns of System 2 decreased steadily over time. The data show that the most rapid settling and densification of the total "MFT + CT" in System 2 occurred in the first 60 d of incubation.

Figures B-14 and B-15 in Appendix B show the $V_{MFT(2)}$ and $V_{CT(2)}$ values in System 2 over 352 d of incubation. The $V_{MFT(2)}$ and $V_{CT(2)}$ were calculated by monitoring the Geo-grid position. The Geo-grid positions in the columns of System 2 were monitored in an in-situ manner. Therefore, the $V_{MFT(2)}$ and $V_{CT(2)}$ data of System 2 were determined without sacrificing the columns.

Figure B-14 shows that the MFT in System 2 expanded slightly in the first 30 d of incubation and started densification thereafter. This trend was similar to the trend observed in the MFT of System 3. In System 2, the largest MFT expansion was 2.9 vol% of $V_{MFT(2)}$ at 14 d of incubation among all six columns. The MFT densified slowly over time and, among the columns of System 2, reached a $V_{MFT(2)}$ range of 0.4 to -1.5 vol% at 49 d of incubation, and

-6.2 to -14.9 vol% at 352 d of incubation. These ranges do not include the data of column C2-2, because column C2-2 had $V_{MFT(2)}$ values deviated greatly from other columns over time. The exact reasons for the large deviation of $V_{MFT(2)}$ in column C2-2 were unknown. The columns in System 2 showed some difference in $V_{MFT(2)}$ values, but the $V_{MFT(2)}$ curve still indicates steady densification of MFT in System 2 with time after an initial expansion in the first 30 d of incubation. By comparison, the $V_{MFT(1)}$ of MFT layer in System 1 showed no obvious trend of densification (see Figure C-1). As in System 1, the $V_{MFT(2)}$ data were also determined by first monitoring the Geo-grid position, which separated the MFT and CT layers in the column. The Geo-grid position in System 2 was measured in in-situ manner without sacrificing the column, whereas the Geo-grid position in System 1 was determined by first sacrificing the column and removing the column cover. This practice of Geo-grid position measurement leads to more accurate Geo-grid position data in System 2 than in System 1. Therefore, the $V_{MFT(2)}$ in System 2 will be more reliable than $V_{MFT(1)}$ in System 1. A conclusion can thus be made that, from the $V_{MFT(2)}$ data of MFT in System 2, the MFT in the CT beneath MFT deposition system (Systems 1 and 2) will consolidate steadily after slight expansion in the first 30 to 60 d of incubation.

Figure B-15 shows that the CT in System 2 consolidated rapidly and to a larger extent than the MFT, similar to the CT in System 1 (see Figure C-1). Most of the $V_{CT(2)}$ decrease occurred in the first 60 d of incubation. The $V_{CT(2)}$ values among the columns in System 2 were between -28.3 and -31.7 vol% at 49 d of incubation, and between -31.8 and -37.4 vol% at 352 d of incubation.

Both the individual tailings volume change ($V_{MFT}$, $V_{CT}$) and solids content can be used to describe the densification behaviour of MFT and CT.

For the MFT in Systems 1 and 2, the $V_{MFT(1)}$ and $V_{MFT(2)}$ are calculated by first measuring the position of Geo-grid. Because the MFT settled and consolidated slowly, the errors caused by the Geo-grid displacement at column filling and by Geo-grid position measurement at column sacrificing will have substantial affect on the accurate calculation of $V_{MFT(1)}$ and $V_{MFT(2)}$. Therefore, the data of $V_{MFT(1)}$ and $V_{MFT(2)}$ should be used with caution. Furthermore, $V_{MFT(2)}$ will likely be more accurate than $V_{MFT(1)}$, due to the more accurate in-situ Geo-grid position monitoring in System 2. The solids content analysis will not be affected by the errors related to the Geo-grid position measurement. Therefore, for the MFT in Systems 1 and 2, it is believed that the solids content data will be more reliable than the tailings volume change ($V_{MFT}$) in characterising the densification behaviour of MFT.

Due to the substantial densification of the CT in both Systems 1 and 2, the $V_{CT(1)}$ and $V_{CT(2)}$ are large values. Thus, the $V_{CT(1)}$ and $V_{CT(2)}$ will be less affected by the errors in Geo-grid position determination. That is, the $V_{CT(1)}$ and $V_{CT(2)}$ are more accurate and reliable than the $V_{MFT(1)}$ and $V_{MFT(2)}$. Therefore, for the CT in Systems 1 and 2, both the solids content and tailings volume change ($V_{CT}$) can be used to characterise the densification behaviour of CT.

As shown in Table 4-5, the columns in System 2 had different chemical amendments ($Na_2SO_4$, $CaSO_4 \cdot 2H_2O$, and $CH_3COONa \cdot 3H_2O$). However, the data of Vtt(2), $V_{MFT(2)}$, and $V_{CT(2)}$ in System 2 over 1 y of incubation showed no

certain effects of these different chemical amendments on the MFT and CT densification rates.

Table 6-4 lists some data of Vtt(2), $V_{MFT(2)}$, and $V_{CT(2)}$ among the columns of System 2, at specific incubation times.

**Table 6-4. Total and individual tailings volume changes at specific incubation times in System 2.**

| $t_{incu}$ (d) | Total and individual tailings volume changes (vol%) | | |
|---|---|---|---|
| | Vtt(2) | $V_{MFT(2)}$ | $V_{CT(2)}$ |
| 49 | -18.1 to -21.5 | 0.4 to -1.5 | -28.3 to -31.7 |
| 77 | -19.8 to -23.5 | -1.1 to -2.6 | -30.5 to -34.3 |
| 133 | -21.0 to -25.7 | -2.9 to -4.8 | -31.9 to -36.3 |
| 352 | -22.5 to -27.7 | -6.2 to -14.9 | -31.8 to -37.4 |

1). For the data of $V_{MFT(2)}$, column C2-2 is not included.

The comparison of $V_{MFT}$ between System 2 (see Figure B-14) and System 3 (see Figure B-2) shows that the MFT in both systems had similar densification over time, and the CT water release had little affect on the densification rate of the overlying MFT in the CT beneath MFT deposition system.

The $V_{CT}$ in Systems 1 and 2 (see Figures C-1 and B-15) had basically the same values as the $V_{CT}$ in System 4 (see Figure B-6). This comparison shows that the overlying MFT layer had no adverse affect on the densification rate of the underlying CT in Systems 1 and 2.

Furthermore, all CT in Systems 1, 2, and 4 showed that most of the settling and densification occurred in the first 60 d of incubation with a slower densification thereafter (see Figures C-1, B-15, and B-6), indicating further densification of CT would be limited.

Tang (1997) discussed two stage CT densification: the first stage of initial settling and the second stage of long-term densification. The initial settling of CT starts immediately after deposition, and usually lasts several days or weeks. Initially, the CT has a strengthened MFT card-house structure within which the coarse sands particles are embedded. During the initial settling of CT, the self-weight force of the sands is transferred to the clay particles and the CT deposit undergoes significant volume reduction as the porewater is pushed out of the clay medium. The second stage of long-term densification begins when the sand grains come in contact with one another and form a sand matrix. The long-term densification of CT proceeds at a much slower rate.

Figure C-1 for System 1, Figure B-15 for System 2, and Figure B-6 for System 4 all show the two stages of densification of CT, that is, the first stage of rapid CT volume reduction which occurred in the first 60 d of incubation, and the second stage of CT densification where the CT consolidated slowly. This settling pattern is matched with the curve of cap water producing rates in Systems 1 and 4 (see Figures B-12 and B-8). The rate of CT dewatering and densification depends on many factors, including the deposit composition, density, and the permeability.

*6.1.3 Cap water volume (Vcw) and cap water producing rate (Rcw)*

6.1.3.1 Cap water volume (Vcw)

Figures B-3, B-7, and B-11 show the Vcw(3), Vcw(4), and Vcw(1) values over 1 y of incubation in Systems 3, 4, and 1, respectively. Comparing Figures B-3, B-7, and B-11, in Systems 1 and 4, most of the cap water was produced in the first 60 d of incubation, and this was matched well with the rapid total tailings volume reduction during the first 60 d of incubation (see Figures B-10 and B-6); whereas in System 3, the cap water was produced over a longer time period. In general, CT released more water than MFT over the 1 y of incubation as shown in Figure B-3 for System 3 and Figure B-7 for System 4.

6.1.3.2 Cap water producing rate (Rcw)

Figures B-4, B-8, and B-12 show the Rcw(3), Rcw(4), and Rcw(1) values over 1 y of incubation in Systems 3, 4, and 1, respectively. System 3 showed a relatively uniform cap water producing rate over the entire incubation period, whereas Systems 1 and 4 showed rapid cap water producing in the first 60 d of incubation.

For System 3 (Cap water/MFT), Figure B-4 shows that no initial rapid cap water producing was observed at the beginning of the incubation. The cap water producing rate was slow at the beginning of incubation and reached a maximum value of approximately 17.0 mL/d at 30 d of incubation. After that time, the cap water producing rate decreased slowly over time and reached 0.8

mL/d at 365 d of incubation. The slow cap water producing rate of MFT was well matched with the slow densification rate of MFT (see Figure B-2).

For System 4 (Cap water/CT), Figure B-8 shows that the cap water producing rate among the columns decreased from approximately 200 mL/d to between 7.5 and 9.4 mL/d at 61 d of incubation, and to approximately 0 mL/d by 212 d of incubation.

For System 1 (Cap water/MFT/CT), Figure B-12 shows that the cap water producing rate among the columns decreased from approximately 200 mL/d during the first day of incubation to between 6.4 and 9.4 mL/d at 62 d of incubation, and to approximately 0 mL/d by 244 d of incubation.

## 6.2 Headspace gas analysis

### *6.2.1 Tedlar bag gas analysis in Systems 1, 3, and 4*

Tedlar bag gas analysis included the Tedlar bag gas volume measurement and the Tedlar bag gas GC analysis. Table B-4 summarises the volume measurement and the GC analysis results of the Tedlar bag gas for the major sacrificed columns in Systems 1, 3, and 4 over 1 y of incubation.

For comparison purpose, Figure C-2 in Appendix C summarises the Tedlar bag gas volume measurements in the three systems over 1 y of incubation. The Tedlar bag gas volume is the gas volume collected in the Tedlar bag of each sacrificed column.

System 3 (Cap water/MFT) showed high and large variation of gas production over time. Over 1 y of incubation, 20 to 2030 mL of Tedlar bag gas from one single column was collected among the sacrificed columns. Visual observations indicated obvious gas evolution from the columns of System 3. Fedorak *et al.* (2000) also reported large variations of Tedlar bag gas volume from Syncrude MFT. The different gas productions and gas holding capacities among MFT columns may cause this gas volume variation from column to column.

Compared with System 3, the Tedlar bag gas volumes in Systems 1 (Cap water/MFT/CT) and 4 (Cap water/CT) were quite low and relatively constant over time (see Table B-4). In System 1, over 1 y of incubation, only 10 to 60 mL of Tedlar bag gas from one single column was collected among the sacrificed columns. In System 4, 17 to 308 mL of Tedlar bag gas from one single column was collected over 1 y of incubation among the sacrificed columns. Visual observations indicated that no obvious gas evolution was seen from columns of Systems 1 and 4.

The Tedlar bag gas GC analysis for Systems 1, 3, and 4 is also presented in Table B-4. The gases identified by the GC analysis included $CH_4$, $N_2$, $O_2$, $CO_2$, $H_2S$, and $C_2H_4$.

For $CH_4$, the Tedlar bag gas from both Systems 1 and 3 showed an increase in $CH_4$ concentration over incubation time. In System 1, the $CH_4$ concentration increased steadily from 0.04 vol% at 29 d of incubation to 4.57 vol% at 213 d of incubation. In System 3, the $CH_4$ concentration increased

steadily from 0.39 vol% at 28 d of incubation to 20.7 vol% at 212 d of incubation, with 34.0 vol% at 334 d of incubation. Fedorak *et al.* (2000) also reported increase trend of $CH_4$ concentration in the headspace of MFT column. However, in some columns of Systems 1 and 3 (including columns C1-8, C1-11, C1-13, C1-14, C1-15, and C1-16 in System 1, columns C3-5, C3-8, C3-9, and C3-12 in System 3), the $CH_4$ concentrations were lower than the level expected from the increasing $CH_4$ concentration trend. These unusually low $CH_4$ concentrations corresponded to high $O_2$ concentrations in the Tedlar bag gas (see Table B-4). The high $O_2$ concentrations indicated column leaking and air intake in the Tedlar bag. This column leaking was also a possible source to cause these low $CH_4$ concentrations, due to the loss of $CH_4$ from the Tedlar bag to the air. The heterogeneity of MFT and CT materials among columns and the errors in GC analysis might also have a role in resulting in the unusually low $CH_4$ concentrations.

In System 4, the $CH_4$ concentrations in the columns were quite low and only between non-detectable concentrations to 0.29 vol% over 1 y of incubation.

Using Tedlar bag gas volume, the headspace volume at column sacrificing, and gas composition (see Table B-4), Figure C-3 summarises the total $CH_4$ volume in the Tedlar bag and column headspace of each sacrificed column in Systems 1, 3, and 4 over 1 y of incubation. By dividing the total $CH_4$ volume by the volume of baseline tailings, the $CH_4$ yield could be calculated. The baseline tailings volume for each system could be calculated based on the deposit dimension at column filling (see Table 4-1). The volume of baseline

tailings was approximately 9.2 L of "MFT+CT" for System 1, 6.1 L of MFT for System 3, and 6.1 L of CT for System 4. Table 6-5 summarises the $CH_4$ yield data.

**Table 6-5. The $CH_4$ yield in Systems 1, 3, and 4.**

| System 1 (Cap water/MFT/CT) | | System 3 (Cap water/MFT) | | System 4 (Cap water/CT) | |
|---|---|---|---|---|---|
| $t_{incu}$ (d) | mL of $CH_4$/L of baseline "MFT+CT" | $t_{incu}$ (d) | mL of $CH_4$/L of baseline MFT | $t_{incu}$ (d) | mL of $CH_4$/L of baseline CT |
| 29 | 0.1 | 28 | 2.8 | 28 | 0.3 |
| 62 | 0.6 | 61 | 2.9 | 61 | 0.4 |
| 91 | 4.9 | 90 | 29.4 | 90 | 0.6 |
| 121 | 3.6 | 120 | 86.7 | 120 | 0 |
| 153 | 0.2 | 152 | 2.0 | 152 | 0.8 |
| 182 | 6.5 | 181 | 140 | 181 | 0.4 |
| 213 | 5.3 | 212 | 142 | 212 | 2.1 |
| 244 | 0.7 | 243 | 5.2 | 243 | 0.9 |
| 267 | 0.2 | 266 | 3.5 | 266 | 0 |
| 303 | 1.0 | 302 | 123 | 302 | 1.0 |
| 335 | 0.1 | 334 | 286 | 334 | 0.3 |
| 366 | 0 | 365 | 46.3 | 365 | 0.5 |

The results presented in Table 6-5 indicate that System 1 yielded more $CH_4$ than System 4, but still less than System 3. Fedorak *et al.* (2000) reported the $CH_4$ yield from Syncrude MFT after about 1 y of incubation was 130 mL of $CH_4$/L of MFT, which was comparable to some $CH_4$ yield data of System 3.

As discussed above, System 3 had higher Tedlar gas volume and $CH_4$ yield than either System 1 or 4. This high $CH_4$ yield can be explained by the activities of methanogens and low sulphate concentrations in the MFT.

Methanogens and SRB are active in Syncrude MFT and CT (Holowenko 2000; Fedorak *et al.* 2000). The methanogens and SRB will compete for substrates. Thermodynamically and kinetically, SRB have the advantage to compete over methanogens. In an anaerobic environment containing sulphate (as is the case in CT), methanogenesis will be inhibited and may be active again after the sulphate has depleted to a certain level. Fedorak *et al.* (2000) noted that both Syncrude MFT and CT have the potential to become methanogenic and methane production occurred substantially after the sulphate concentration has dropped to about 20 mg/L.

Figures C-4, C-5, and C-6 show the sulphate concentrations in the cap water and porewaters of MFT and CT in Systems 1, 3, and 4 over 1 y of incubation, respectively. It is clear that, in Systems 1 and 4, the $SO_4^{2-}$ concentrations in most of the cap water and porewater were well above 20 mg/L over 1 y of incubation. Accordingly, the high concentrations of $SO_4^{2-}$ in the cap water and porewater samples will favour the activity of SRB and inhibit the methane production. In System 3, however, the $SO_4^{2-}$ concentrations in most of the cap water and porewater samples were well below 20 mg/L, thus favouring potential methanogenesis and methane production.

Although high concentrations of $SO_4^{2-}$ will inhibit methane production, methane production does not always require low $SO_4^{2-}$ concentrations (Fedorak *et al.* 2002). This observation may provide an explanation for the fact that in the Tedlar bag gases of Systems 1 and 4, there was still some measurable $CH_4$. With the decrease in $SO_4^{2-}$ concentration, the methane production increased in System

1 (see Table B-4). System 4 had low methane production over the 1 y of incubation (see Table B-4), which was consistent with its high $SO_4^{2-}$ concentration over that time period (see Figure C-6).

As shown in Table B-4, the main gas present in the Tedlar bag was $N_2$. Because the nitrate concentrations in all the cap waters and porewaters in Systems 1, 3, and 4 were low and mostly below the detection limits (<0.5 mg/L), it is unlikely that this high volume of $N_2$ was from the denitrification process. The most possible $N_2$ source was from the $N_2$ flushing at column filling, because all columns were flushed with $N_2$ before and during column filling for several minutes.

The air contains approximately 78 vol% $N_2$, 21 vol% $O_2$, and 0.03 vol% $CO_2$ (Hopp and Hennig 1983). The constant presence of $O_2$ in the Tedlar bag gas is believed to be mainly from air, due to either the incomplete $N_2$ flushing at column filling or the leaking of columns or Tedlar bags during incubation. Some columns (i.e. columns C1-4, C1-8, C1-13, C1-14, C1-15, C3-5, C3-8, C3-9, and C4-9) had approximately 20 vol% $O_2$ (see Table B-4), essentially the same $O_2$ concentration as in air. This high $O_2$ concentration may indicate severe column leaking during incubation. However, the sampling during GC analysis may also introduce $O_2$ into the results if handled improperly. Most of other columns maintained fairly low concentrations of $O_2$, indicating a good column sealing and anaerobic environment (see Table B-4).

Figure C-7 shows the $CO_2$ concentrations in the headspace of Systems 1, 3, and 4 over 1 y of incubation. It can be seen that, in the early incubation, the

$CO_2$ concentrations in Systems 1, 3, and 4 increased with time and reached 4 to 8 vol% in 180 d of incubation. This increase in headspace $CO_2$ concentration was likely from microbial activity that occurred in the MFT and CT. As stated in the literature (Maier 2000), in the process of microbial metabolism, the heterotrophic bacteria will utilise the naturally occurring organic materials as substrates and produce $CO_2$ in both aerobic and anaerobic environments. Under aerobic conditions, the heterotrophic bacterial activity will transform the carbon in the substrate to about 50% of $CO_2$ and 50% of new cell mass. Under anaerobic conditions, a smaller portion of the carbon is used for new cell mass and a larger portion goes to $CO_2$ and $CH_4$. Thus, the $CO_2$ in the Tedlar bag gas is believed to be the result of and an indicator of active microbial activity (either aerobic or anaerobic) occurring in the MFT and CT. The unusually low $CO_2$ concentrations in some columns (see Figure C-7) are likely due to the dilution by air or loss from leaking. This is supported by the $O_2$ analysis, which indicated generally high $O_2$ concentrations in those columns with unusually low $CO_2$ concentrations (see Table B-4).

The $CO_2$ concentrations in the Tedlar bag should be the same as in the column headspace. During long incubation time, the $CO_{2(g)}$ concentrations in the headspace gas are deemed to be in equilibrium with the $CO_{2(aq)}$ concentrations in the cap water, which is in direct contact with the headspace gas. Thus, Henry's law might be used to calculate the $CO_{2(g)}$ concentrations in the headspace gas based on the concentrations of $CO_{2(aq)}$ in the cap water. In applying the Henry's

law for the $CO_{2(g)}$ concentration calculations in the headsapce gas, the following Equations 6-1 to 6-6 are used (Sawyer, *et al.* 1994; Snoeyink and Jenkins 1980):

$$H_2CO_3^* \rightleftharpoons H^+ + HCO_3^- \quad \text{.....(6-1)}$$

$$[H_2CO_3^*] = ([H^+]\times[HCO_3^-])/K_{A1} = ([H^+]\times[HCO_3^-])/(4.3\times10^{-7}) \quad \text{.....(6-2)}$$

$$[H_2CO_3^*] = [CO_{2(aq)}] + [H_2CO_{3(aq)}] \quad \text{.....(6-3)}$$

$$[H_2CO_3^*] \approx [CO_{2(aq)}] \quad \text{.....(6-4)}$$

$$CO_{2(aq)} \rightleftharpoons CO_{2(g)} \quad \text{.....(6-5)}$$

$$K_H = 1.0\times10^{-1.5} = [CO_{2(aq)}]/Pco_{2(g)} \approx [H_2CO_3^*]/Pco_{2(g)} \quad \text{.....(6-6)}$$

where $K_H$ is the Henry's law constant at room temperature; $Pco_{2(g)}$ is the partial pressure of $CO_2$ in the headspace gas with the unit of atm.

Thus, with the known concentrations of $HCO_3^-$ and pH in the cap water at room temperature, by using the Equations 6-1 through 6-6, the partial pressure of $CO_2$ in the headspace gas can be calculated.

For System 1, Table C-1 in Appendix C provides a comparison of the measured $CO_2$ concentrations by GC and the calculated $CO_2$ concentrations

using Henry's law in the headspace. It can be seen that the calculated $CO_2$ concentrations in the headspace gas are comparable to the measured $CO_2$ concentrations in the headspace gas. The calculation of the $CO_2$ concentrations using the above equations confirms the results of measured $CO_2$ concentrations in the headspace gas or Tedlar bag gas.

As shown in Table B-4, in some columns, low concentrations (for the most part, <1.0 vol%) of $H_2S$ and $C_2H_4$ were detected in the headspace. Both the $H_2S$ and $C_2H_4$ are assumed to be the products of microbial activity, as detailed below.

As the result of SRB activity, sulphate will be reduced to sulphide. Depending on the pH of the environment, the sulphide will predominantly exist either as $H_2S$ when pH<7.3 or as $HS^-$ when pH>7.3 (Fedorak *et al.* 2000). As shown in Table B-7 for System 3, Table B-8 for System 4, and Table B-9 for System 1, most of the pH values in the cap water and tailings porewater in all three systems were between 7.6 to 8.3. This pH range will produce $HS^-$ as the predominant form of sulphide. The $HS^-$ will further most likely combine with heavy metals to precipitate as sulphide-containing minerals, also called acid volatile sulphides. The rare detection and low concentration of $H_2S$ in column headspace was well matched with the alkaline pH range of 7.6 to 8.3. Furthermore, System 1 had similar $H_2S$ release as in Systems 3 and 4.

Ethylene has been commonly found in the Syncrude MFT. Ethylene has adverse effects on plant growth. The presence of ethylene was attributed to the microbial activity of either aerobic or anaerobic microorganisms. (Fedorak *et al.*

2000). In this project, ethylene was occasionally detected in the headspace with concentrations between 9 and 3000 ppm by volume (see Table B-4).

*6.2.2 In-situ headspace gas analysis in System 2*

For the headspace gas analysis in System 2, the Micro-GC was directly connected to the headspace of columns via a piece of stainless steel tubing (see Section 4.2.2). Therefore, the headspace gas analysis in System 2 was performed in an in-situ manner to regularly monitor the headspace gas. Because any evolved headspace gas will quickly disperse into Tedlar bag, the results of in-situ headspace gas analysis should be the same as in the Tedlar bag gas analysis.

As discussed previously, in-situ cap water sampling was performed each time after completing the in-situ headspace gas analysis. The cap water was completely removed at each sampling. The void in the headspace created by removing the cap water was filled with pure $N_2$. This increased headspace volume and introduction of $N_2$ will dilute and decrease the concentrations of existing headspace gases (other than $N_2$), as compared to Tedlar bag gas analysis. Also, the frequent in-situ headspace gas analysis and cap water sampling could cause more air contamination in the headspace, thus leading to a higher $O_2$ concentration.

The headspace volume increased with each cap water sampling. The increased headspace volume can be estimated from the total tailings volume change (Vtt). Figure B-13 shows the Vtt(2) values with time in System 2. The

Vtt(2) is defined in Equation 4-2. At time 0, the columns in System 2 contained approximately an 81-cm height of "MFT+CT" and a 10-cm height of headspace. At 133 d of incubation (i.e. the 9$^{th}$ sampling), the six columns in System 2 had a Vtt(2) in the range of -21.0 to –26.0% (see Figure B-13). This level of Vtt(2) translates into a headspace volume 2.7 to 3.1 times that of the original headspace volume. At 352 d of incubation (i.e. the 10$^{th}$ sampling), the headspace volume in System 2 had increased even further.

For columns in System 2, there were 10 samplings for the in-situ headspace gas analysis over 1 y of incubation, as shown in Table B-5. In comparison to the Tedlar bag gas analysis in System 1 (see Table B-4), the in-situ headspace gas analysis in System 2 showed constantly low $CH_4$ and $CO_2$ concentrations, and a high $O_2$ concentration. The low $CH_4$ and $CO_2$ concentrations may be partially due to the dilution effect caused by the increased headspace volume and newly added $N_2$ gas with each cap water sampling and removal. As discussed above, at 133 d of incubation, the headspace volume in the six columns of System 2 was about 3 times the original headspace volume. It should be noted that the Tedlar bags in System 2 had little gas collected over time; therefore all of the gases were basically contained in the headspace. If the in-situ headspace $CH_4$ and $CO_2$ concentrations were multiplied by a factor of 3 to take into account the increased headspace, the $CO_2$ concentrations in System 2 had similar values as those in Tedlar bag gas of System 1, but the $CH_4$ concentrations still showed lower values than those in System 1. That is, the $CH_4$ yield in System 2 was lower than in System 1. The $O_2$ concentration in

System 2 increased with each sampling, indicating some possible contamination by air during headspace gas analysis and cap water sampling.

In the headspace of System 2, some low concentrations (<1.0 vol%) of $H_2S$ and $C_2H_4$ were occasionally detected. As discussed above, both the $H_2S$ and $C_2H_4$ are believed to be the products of microbial activity.

In characterising the evolved gases in Tedlar bag or headspace from the MFT and CT deposits, due to the interference and contamination caused by the frequent in-situ cap water sampling and headspace gas analysis, the in-situ headspace GC analysis in System 2 would appear to be less accurate than the Tedlar bag gas analysis in Systems 1, 3, and 4.

As mentioned above, the columns in System 2 had different chemical amendments in the CT at column filling (see Table 4-5). The in-situ headspace gas analysis of System 2 showed that the six columns in System 2 had similar levels of $CH_4$, $CO_2$, and $H_2S$ detection, indicating that the different chemical amendments added to the CT had little effect on the headspace gas evolution and gas composition during 1 y of incubation.

### 6.3 Physical and chemical characteristics of cap water, MFT, and CT

Both MFT and CT are heterogeneous materials, so the physical, chemical, and microbiological data obtained from one location could vary to

some degree from the data obtained from other locations. Caution should be used in interpreting the data.

### *6.3.1 Physical and chemical characteristics in System 3 (Cap water/MFT)*

#### 6.3.1.1 Calcium and magnesium concentrations and the pH

According to Table B-6, the baseline $Ca^{2+}$ concentration in MFT for System 3 (Base MFT 2) was 23 mg/L. Figure C-8 shows the $Ca^{2+}$ concentrations in the cap water and MFT porewater of System 3 over 1 y of incubation. Both the cap water and MFT porewater in System 3 had quite constant $Ca^{2+}$ concentrations over time, with the $Ca^{2+}$ concentrations in the cap water ranging from 19 to 27 mg/L and the $Ca^{2+}$ concentrations in the MFT porewater ranging from 15 to 21 mg/L over 1 y of incubation. These $Ca^{2+}$ concentrations in the cap water and MFT porewater are close to 23 mg/L of the baseline $Ca^{2+}$ concentration in the MFT, indicating that the MFT released $Ca^{2+}$ without $Ca^{2+}$ concentration abatement and that the $Ca^{2+}$ release was stable over this incubation period. The upper and lower zones of MFT showed similar $Ca^{2+}$ concentrations over time. The cap water had a consistently higher $Ca^{2+}$ concentration than the MFT porewater over 1 y of incubation.

According to Table B-6, the baseline $Mg^{2+}$ concentration in MFT for System 3 (Base MFT 2) was 16 mg/L. Figure C-9 shows the $Mg^{2+}$ concentration in System 3 over 1 y of incubation. The $Mg^{2+}$ concentrations in the cap water

and MFT porewater were constant over time. The $Mg^{2+}$ concentrations in the cap water ranged from 13 to 18 mg/L, while the $Mg^{2+}$ concentrations in the MFT porewater ranged from 11 to 15 mg/L over 1 y of incubation. The $Mg^{2+}$ concentrations in the cap water and MFT porewater were close to 16 mg/L of the baseline $Mg^{2+}$ concentration in MFT, indicating that the MFT released $Mg^{2+}$ without $Mg^{2+}$ concentration abatement and that the $Mg^{2+}$ release was stable over this incubation period. The upper and lower zones of MFT showed similar $Mg^{2+}$ concentrations over time. The cap water had a consistently higher $Mg^{2+}$ concentration than the MFT porewater over 1 y of incubation.

Figure C-10 shows that the pH of cap water and MFT porewater were similar, and for the most part within the range of 7.7 to 8.3 over 1 y of incubation.

6.3.1.2 Sulphate concentration and methanogenesis

According to Table B-6, the baseline $SO_4^{2-}$ concentration in MFT for System 3 (Base MFT 2) was 14 mg/L. Figure C-5 shows the $SO_4^{2-}$ concentration in System 3 over 1 y of incubation. In both the cap water and MFT porewater, $SO_4^{2-}$ concentrations decreased with time and reached a non-detectable level (<0.5 mg/L) after the first 90 d of incubation. Then the $SO_4^{2-}$ concentrations in both the cap water and MFT porewater remained at non-detectable levels (<0.5 mg/L) until 243 d of incubation. These results may indicate that the SRB were still active under initially low $SO_4^{2-}$ concentration and could reduce $SO_4^{2-}$ in MFT. The reduction of the initially low concentration

of $SO_4^{2-}$ in MFT will lead to a slight $HCO_3^-$ concentration increase, as shown in Equation 2-2. A slight increase in $HCO_3^-$ concentration over 1 y of incubation was observed (see Figure C-11). The $SO_4^{2-}$ concentrations in the cap water at late incubation time were in the range of 10 to 11 mg/L. No certain explanations can be found to account for this increase in $SO_4^{2-}$ concentration in the cap water.

Table B-7 lists the $S^{2-}$ analysis results on the cap water and MFT porewater in System 3. All the $S^{2-}$ concentrations in the cap water and MFT porewater were non-detectable (<0.05 mg/L). These essentially zero values may reflect the low quantity of $S^{2-}$ resulting from reduction of initially a low concentration of $SO_4^{2-}$ by SRB. It is also possible that the sulphide was trapped as minerals (e.g. FeS) in MFT.

As discussed previously in Section 2.4, with low $SO_4^{2-}$ concentration (<20 mg/L), the condition in MFT will favour the methanogen activity when the environment becomes anaerobic. Figure C-12 shows the redox potential in the MFT of System 3 over 1 y of incubation. The suitable Eh for methanogen activity is in the range of –150 to –220 mV (Fedorak *et al.* 2000). Figure C-12 shows that, over 1 y of incubation, the Eh in MFT was mostly in the range of 21 to 77 mV. These high Eh values in MFT were unexpected. But the consistent Eh values in MFT may in some way justify the measurement results. The positive Eh in the MFT of System 3 should not favour the methanogen activity. However, the Tedlar bag gas analysis showed that high volume of $CH_4$ was detected in the headspace of System 3 (see Figure C-3). The trapped gas analysis in the MFT of System 3 also showed that $CH_4$ was detected in most of the MFT samples (see

Table B-12). Therefore, the methanogenesis was occurring in the MFT of System 3, despite the positive Eh values. Fedorak *et al.* (2000) suggested the presence of anaerobic microsites in the MFT and CT materials. The bulk Eh measurement may be not sensitive enough to the Eh in these anaerobic microsites, while the anaerobic microsites may have negative enough Eh for methanogensis to occur.

#### 6.3.1.3 Solids content

Figure C-13 shows the solids content in the MFT of System 3 over 365 d of incubation. The solids content of MFT in System 3 showed a steady increase over this incubation period. This trend matches the steady water release from the MFT of System 3 (see Figure B-3). Over the 365 d of incubation, the solids content increased from baseline data of 38.0 wt% (Base MFT 2) to 44.7 wt% in the upper MFT zone and 45.6 wt% in the lower MFT zone. The upper and lower MFT zones showed similar solids content over time, indicating uniform densification throughout the MFT layer.

### *6.3.2 Physical and chemical characteristics in System 4 (Cap water/CT)*

#### 6.3.2.1 Bromide tracer analysis

The tracer $Br^-$ was added to the CT at column filling in the form of NaBr. Approximately 0.22 g of NaBr was added to 1 L CT at column filling for

System 4 (see Table 4-3). The baseline $Br^-$ concentration in the CT for System 4 (Base CT 3) was 260 mg/L (see Table B-6). This measured $Br^-$ concentration of 260 mg/L in the baseline CT for System 4 is verified to be close to 258 mg/L. The figure 258 mg/L is the calculated $Br^-$ concentration based on the planned NaBr dosage (Table 4-3). The verification will be presented at the end of this section.

Figure C-14 shows the $Br^-$ concentration in the cap water and CT porewater of System 4 over 1 y of incubation. Figure C-14 shows that, over 1 y of incubation, the $Br^-$ concentrations in the cap water and CT porewater were quite constant and in the range of 250 to 280 mg/L, close to 260 mg/L of the baseline $Br^-$ concentration. The constant $Br^-$ concentration at values close to the baseline data over 1 y of incubation shows that $Br^-$ is a stable tracer in the CT material.

A mass balance of $Br^-$ in the whole column of System 4 was attempted. System 4 contains CT only at time 0, and cap water plus CT during incubation. The $Br^-$ concentrations in the cap water and CT porewater were measured for each sacrificed column in System 4 (Table B-8). By assuming that the $Br^-$ is only present in the water (cap water and porewater), if the cap water and porewater volumes are known, the mass balance of $Br^-$ in System 4 can be achieved. The cap water volume in each sacrificed column of System 4 can be calculated by using the absolute height of cap water at column sacrificing (see Table B-2) and the column ID. The column ID is 12.1 cm. The porewater volume can be determined from an empirical equation that relates the density of

MFT or CT materials to their solids contents, as shown in Equation 6-7 (Dr. MacKinnon, Syncrude Canada Ltd., personal communication):

$$\rho = (1/(1-0.0062 \times wt\%)) \times 1000 \quad \text{.....(6-7)}$$

where $\rho$ is the density of MFT or CT (g/L of MFT or CT); wt% is the solids contents of MFT or CT.

From Equation 6-7, an estimation of the masses of solids and porewater per unit volume of MFT or CT as a function of solids content can be established. Table C-2 provides the estimations of the masses of solids and porewater per unit volume of MFT or CT at specific solids content. In establishing Table C-2, it is assumed that the total mass of MFT or CT is the sum of the mass of solids and the mass of porewater. Therefore, once the CT volume is known, the porewater volume can be determined by using Table C-2.

For each sacrificed column in System 4, the CT volumes at time 0 (baseline CT) and at column sacrificing can be calculated from the absolute heights of CT data listed in Table B-2 and the column ID. Thus, the porewater volume of CT can be obtained by together using Table C-2.

With the known $Br^-$ concentrations in each water (cap water and porewater, see Table B-8) and the calculated cap water and porewater volumes, the mass of $Br^-$ in the cap water and porewater can be calculated. Table 6-6 summarises the $Br^-$ mass calculations in the baseline CT (Base CT 3), and in the

cap water and CT porewater from the sacrificed columns of System 4. It is seen that over 1 y of incubation, the $Br^-$ recovery (relative to the $Br^-$ mass at time 0) in System 4 was in the range of 93 to 104%. This mass recovery indicates that the $Br^-$ is a stable and conservative tracer in CT material over the experiment time.

It should be noted that, based on the planned NaBr dosage of 0.22 g/L of CT in the baseline CT of System 4 (Table 4-3) and the typical CT volume of 6.1 L at time 0 (equivalent to a height of 54 cm in the column), the total mass of $Br^-$ in the CT of each column of System 4 at time 0 is approximately 1053 mg, and the calculated baseline $Br^-$ concentration in the CT of System 4 at time 0 is 258 mg/L. Here, 1053 mg $=(84/(23+84))\times0.22\times6.1\times1000$ mg, and 258 mg/L $=1053/(0.67\times6.1)$ mg/L, where 6.1 L is the volume of CT at time 0; 23 and 84 are atomic weights of sodium and bromine, respectively; 0.67 is the volume (L) of porewater per L of CT at solids content of 56.6 wt% (in Base CT 3) and is from Table C-2. The figure 1053 mg is quite close to the $Br^-$ mass at time 0 obtained from the $Br^-$ concentrations (see Table 6-6), and the figure 258 mg/L is quite close to the measured baseline $Br^-$ concentration of 260 mg/L in the CT of System 4 (see Table B-6). This comparison in one way justifies the methods and calculation results of $Br^-$ mass listed in Table 6-6, and also shows that the $Br^-$ is a stable tracer in the CT material.

**Table 6-6. The $Br^-$ mass balance calculation in System 4.**

| Column | $t_{incu}$ (d) | Total mass of $Br^-$ in the whole CT at time 0 (mg) | Mass of $Br^-$ at column sacrificing (mg) | | | | $Br^-$ mass recovery (%) |
|---|---|---|---|---|---|---|---|
| | | | In cap water | In CT-U | In CT-L | Total mass of $Br^-$ in the whole column | |
| C4-1 | 28 | 1085 | 379 | 397 | 347 | 1123 | 104 |
| C4-2 | 61 | 1083 | 469 | 313 | 302 | 1084 | 100 |
| C4-3 | 90 | 1081 | 474 | 293 | 280 | 1047 | 97 |
| C4-4 | 120 | 1083 | 502 | 287 | 274 | 1063 | 98 |
| C4-5 | 152 | 1083 | NA | 287 | 259 | NA | NA |
| C4-6 | 181 | 1075 | 511 | 277 | 261 | 1049 | 98 |
| C4-7 | 212 | 1079 | 494 | 252 | 274 | 1020 | 94 |
| C4-8 | 243 | 1081 | 508 | 265 | 272 | 1045 | 97 |
| C4-9 | 266 | 1079 | 534 | 289 | 277 | 1100 | 102 |
| C4-10 | 303 | 1083 | 534 | 290 | 278 | 1102 | 102 |
| C4-11 | 334 | 1079 | 514 | 255 | 247 | 1016 | 94 |
| C4-12 | 365 | 1083 | 511 | 251 | 248 | 1010 | 93 |

1). $Br^-$ mass recovery (%) =((total mass of $Br^-$ at column sacrificing)/(total mass of $Br^-$ at time 0))×100%.
2). NA=not available.

6.3.2.2 Calcium and magnesium concentrations and the pH

According to Table B-6, the baseline $Ca^{2+}$ concentration in CT for System 4 (Base CT 3) was 82 mg/L. Figure C-15 shows the $Ca^{2+}$ concentration in System 4 over 1 y of incubation.

In the CT, Figure C-15 shows that most of the decrease in $Ca^{2+}$ concentration occurred during the first 60 d of incubation. At 61 d of incubation, the $Ca^{2+}$ concentration in CT decreased dramatically from the baseline concentration of 82 mg/L to 26 mg/L in CT-U and 25 mg/L in CT-L. After that time, the $Ca^{2+}$ concentration was quite stable and in the range of 18 to 27 mg/L until the end of 365 d of incubation. The upper and lower zones of CT had similar $Ca^{2+}$ concentrations over 1 y of incubation.

In the cap water, Figure C-15 shows that the $Ca^{2+}$ concentration was in the range of 20 to 68 mg/L over 1 y of incubation, higher than in the CT porewater. The higher $Ca^{2+}$ concentration in the cap water may be due in part to the pH difference. Figure C-16 shows that the cap water had a lower pH than the CT porewater. The lower pH in the cap water may contribute to a higher $Ca^{2+}$ concentration, due to the effect of redissolution of $CaCO_3$ at lower pH conditions. Compared to the CT porewater, the cap water showed a large fluctuation of $Ca^{2+}$ concentration over time (Figure C-15).

Using the same procedures as for the $Br^-$ mass balance calculations in Section 6.3.2.1, a mass balance of $Ca^{2+}$ in the whole column of System 4 was

attempted. Table 6-7 lists the results of $Ca^{2+}$ mass balance calculations in System 4.

It can be seen from Table 6-7 that, over 1 y of incubation, only less than 50% of the original $Ca^{2+}$ mass still exists in the water, suggesting that more than 50% of the original $Ca^{2+}$ mass was removed from the water by some mechanisms, as detailed below.

The mechanisms leading to a change in $Ca^{2+}$ concentration in System 4 may include ion exchange between $Ca^{2+}$ and $Na^{+}$ on the clays of CT, and precipitation or redissolution of $CaCO_3$ at high or low pH conditions.

Ion exchange between $Ca^{2+}$ and $Na^{+}$ on the clay surface will cause a change in $Na^{+}$ concentration, leading to an increase in $Na^{+}$ concentration in the porewater. Figure C-17 shows the $Na^{+}$ concentration in System 4 over 1 y of incubation. In the early incubation, Figure C-15 shows a rapid decrease in the $Ca^{2+}$ concentration in CT. This decrease in $Ca^{2+}$ concentration was not accompanied by an increase in the $Na^{+}$ concentrations of either the cap water or CT porewater during this period (see Figure C-17). This result indicates that ion exchange between $Ca^{2+}$ and $Na^{+}$ in CT most likely did not occur. The slight increase in the $Na^{+}$ concentrations of the cap water and CT porewater at late incubation times may not result from the ion exchange between $Ca^{2+}$ and $Na^{+}$, for the reason that the $Ca^{2+}$ concentration in the CT porewater was quite constant during this late incubation period (see Figure C-15).

**Table 6-7. The $Ca^{2+}$ mass balance calculation in System 4.**

| Column | $t_{incu}$ (d) | Total mass of $Ca^{2+}$ in the whole CT at time 0 (mg) | Mass of $Ca^{2+}$ at column sacrificing (mg) | | | | $Ca^{2+}$ mass recovery (%) |
|---|---|---|---|---|---|---|---|
| | | | In cap water | In CT-U | In CT-L | Total mass of $Ca^{2+}$ in the whole column | |
| C4-1 | 28 | 342 | 84 | 50 | 44 | 178 | 52 |
| C4-2 | 61 | 342 | 60 | 32 | 29 | 121 | 35 |
| C4-3 | 90 | 341 | 74 | 26 | 24 | 124 | 37 |
| C4-4 | 120 | 342 | 97 | 30 | 28 | 155 | 46 |
| C4-5 | 152 | 342 | NA | 26 | 24 | NA | NA |
| C4-6 | 181 | 339 | 109 | 29 | 25 | 163 | 48 |
| C4-7 | 212 | 340 | 62 | 22 | 24 | 108 | 32 |
| C4-8 | 243 | 341 | 38 | 24 | 24 | 86 | 25 |
| C4-9 | 266 | 340 | 71 | 22 | 18 | 111 | 33 |
| C4-10 | 303 | 342 | 88 | 26 | 23 | 137 | 40 |
| C4-11 | 334 | 340 | 134 | 26 | 22 | 182 | 54 |
| C4-12 | 365 | 342 | 113 | 25 | 24 | 162 | 47 |

1). $Ca^{2+}$ mass recovery (%) =((total mass of $Ca^{2+}$ at column sacrificing)/(total mass of $Ca^{2+}$ at time 0))×100%.

2). NA=not available.

Therefore, the major mechanism causing the reduction in $Ca^{2+}$ concentration in the CT of System 4 in the first 60 d of incubation may be due to the precipitation of $CaCO_3$. $CaCO_3$ has solubility product (Ksp) of $8.7\times10^{-9}$ at 25°C (Nazaroff and Alvarez-Cohen 2001; AWWA 1999). At alkaline conditions, $Ca^{2+}$ will precipitate as $CaCO_3$ if the ratio of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp is greater than 1, according to the following reaction:

$$Ca^{2+} + CO_3^{2-} \rightleftharpoons CaCO_{3(s)} \quad \text{.....(6-8)}$$

This calcium carbonate precipitation usually takes place before pH level has exceeded 10 (Sawyer *et al.* 1994). The pH in the cap water and CT porewater of System 4 was within 7.7 to 8.2 (see Figure C-16), showing alkaline conditions. Because of the high concentration of $Ca^{2+}$ and alkaline pH in the CT porewater, the reduction in $Ca^{2+}$ concentration in the CT of System 4 might be due to this $CaCO_3$ precipitation.

Table 6-8 summarises the ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in each zone of System 4 over 1 y of incubation, where $[Ca^{2+}]$ and $[CO_3^{2-}]$ are the molar concentrations (M) of $Ca^{2+}$ and $CO_3^{2-}$ in the cap water and porewater of System 4; Ksp is the solubility product of $CaCO_3$. The Ksp is $8.7\times10^{-9}$ at 25°C (Nazaroff and Alvarez-Cohen 2001; AWWA 1999).

The calculation of this ratio of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp is as follows. From the concentration of $HCO_3^-$, the concentration of $CO_3^{2-}$ can be calculated based

on the Equations 6-9 and 6-10 (Sawyer, *et al.* 1994). Furthermore, the molar concentrations of $[H^+]$, $[HCO_3^-]$, and $[Ca^{2+}]$ (M) can be converted from the concentrations (mg/L) provided in Table B-8. Thus, the ratio of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp can be obtained for System 4.

$$HCO_3^- \rightleftharpoons H^+ + CO_3^{2-} \quad \text{.....(6-9)}$$

$$K_{A2}=([H^+]\times[CO_3^{2-}])/[HCO_3^-]=4.69\times10^{-11} \quad \text{.....(6-10)}$$

Table 6-8 shows that the ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in all zones of System 4 over 1 y of incubation are in the range of 4 to 13, indicating the precipitation of $CaCO_3$ is a possible route for the removal of $Ca^{2+}$ in System 4.

**Table 6-8. The ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in the cap water and porewaters of System 4 at column sacrificing.**

| Column | $t_{incu}$ (d) | The ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp at column sacrificing | | |
|---|---|---|---|---|
| | | In cap water | In CT-U | In CT-L |
| C4-1 | 28 | 6 | 12 | 12 |
| C4-2 | 61 | 10 | 7 | 6 |
| C4-3 | 90 | 9 | 7 | 7 |
| C4-4 | 120 | 7 | 8 | 9 |
| C4-5 | 152 | | 6 | 7 |
| C4-6 | 181 | 8 | 7 | 7 |
| C4-7 | 212 | 7 | 6 | 9 |
| C4-8 | 243 | 6 | 7 | 4 |
| C4-9 | 266 | 6 | 4 | 5 |
| C4-10 | 303 | 5 | 6 | 7 |
| C4-11 | 334 | 13 | 9 | 10 |
| C4-12 | 365 | 8 | 9 | 9 |

Figure C-18 shows the $Mg^{2+}$ concentration in System 4 over 1 y of incubation. The CT for System 4 (Base CT 3) had a baseline $Mg^{2+}$ concentration of 30 mg/L.

In the CT porewater of System 4, similar to the situation of $Ca^{2+}$, the $Mg^{2+}$ concentration decreased most rapidly in the first 60 d of incubation and reached 13 mg/L in both CT-U and CT-L at 61 d of incubation. After that time, the $Mg^{2+}$ concentration in CT porewater was stable and in the range of 11 to 14 mg/L for the remainder of the experiment. Both the upper and lower zones of CT had similar $Mg^{2+}$ concentrations.

In the cap water of System 4, the $Mg^{2+}$ concentration was quite constant and in the range of 22 to 30 mg/L over 1 y of incubation (see Figure C-18).

6.3.2.3 Sulphate concentration

Figure C-6 shows the $SO_4^{2-}$ concentration in System 4 over 1 y of incubation. According to Table B-6, the baseline $SO_4^{2-}$ concentration in the CT for System 4 (Base CT 3) was 1100 mg/L.

In the CT of System 4, Figure C-6 shows that the most decrease in $SO_4^{2-}$ concentration occurred in the first 60 d of incubation. At 61 d of incubation, the $SO_4^{2-}$ concentration in the CT decreased dramatically to 497 mg/L in CT-U and 413 mg/L in CT-L. After that time, the $SO_4^{2-}$ concentration showed a slow decrease and reached 259 mg/L in CT-U and 178 mg/L in CT-L at 365 d of incubation. The upper and lower zones of CT had similar $SO_4^{2-}$ concentrations over 1 y of incubation.

In the cap water of System 4, Figure C-6 shows that the $SO_4^{2-}$ concentration was in the range of 490 to 1070 mg/L over 1 y of incubation, higher than in the CT porewater.

Using the same procedures as for the $Br^-$ mass balance calculations in Section 6.3.2.1, a mass balance of $SO_4^{2-}$ in the whole column of System 4 was attempted. Table 6-9 lists the results of $SO_4^{2-}$ mass balance calculations in System 4.

It can be seen from Table 6-9 that, after 1 y of incubation, only less than 50% of the original $SO_4^{2-}$ mass still exits in the water, suggesting that more than 50% of the original $SO_4^{2-}$ mass was removed from the water by some mechanisms. As discussed previously in Section 2.4, with the high $SO_4^{2-}$ concentration, the conditions in the CT of System 4 should favour $SO_4^{2-}$ reduction by SRB when the environment becomes anaerobic.

The decrease in $SO_4^{2-}$ concentration in the CT porewater of System 4 over time most likely indicates the SRB activity. As discussed in Section 2.4, the activity of SRB will produce $HCO_3^-$ (according to Equation 2-2). Figure C-19 shows the $HCO_3^-$ concentration in System 4 over incubation time, indicating a steady $HCO_3^-$ concentration increase in both the cap water and porewater of the CT over incubation time. In CT porewater, the increase in $HCO_3^-$ concentration was well matched with the decrease in $SO_4^{2-}$ concentration (see Figure C-6), supporting $SO_4^{2-}$ reduction by SRB in the CT layer.

**Table 6-9. The $SO_4^{2-}$ mass balance calculation in System 4.**

| Column | $t_{incu}$ (d) | Total mass of $SO_4^{2-}$ in the whole CT at time 0 (mg) | Mass of $SO_4^{2-}$ at column sacrificing (mg) | | | | $SO_4^{2-}$ mass recovery (%) |
|---|---|---|---|---|---|---|---|
| | | | In cap water | In CT-U | In CT-L | Total mass of $SO_4^{2-}$ in the whole column | |
| C4-1 | 28 | 4591 | 1500 | 1145 | 848 | 3493 | 76 |
| C4-2 | 61 | 4583 | 1413 | 598 | 480 | 2491 | 54 |
| C4-3 | 90 | 4574 | 1972 | 400 | 410 | 2782 | 61 |
| C4-4 | 120 | 4583 | 1790 | 181 | 79 | 2050 | 45 |
| C4-5 | 152 | 4583 | NA | 446 | 352 | NA | NA |
| C4-6 | 181 | 4549 | 1716 | 386 | 64 | 2166 | 48 |
| C4-7 | 212 | 4566 | 1388 | 291 | 314 | 1993 | 44 |
| C4-8 | 243 | 4574 | 957 | 261 | 272 | 1490 | 33 |
| C4-9 | 266 | 4566 | 1510 | 384 | 313 | 2207 | 48 |
| C4-10 | 303 | 4583 | 1408 | 242 | 198 | 1848 | 40 |
| C4-11 | 334 | 4566 | 1908 | 310 | 164 | 2382 | 52 |
| C4-12 | 365 | 4583 | 1637 | 250 | 170 | 2057 | 45 |

1). $SO_4^{2-}$ mass recovery (%) =((total mass of $SO_4^{2-}$ at column sacrificing)/(total mass of $SO_4^{2-}$ at time 0))×100%.
2). NA=not available.

Table B-8 shows that the Eh in the CT of System 4 was quite constant and in the range of –168 to –217 mV over 1 y of incubation. This Eh range should favour both SRB and methanogen activity. However, the high $SO_4^{2-}$ concentration may inhibit methanogen activity. The trapped gas analysis on the CT samples of System 4 (see Table B-12) shows that trapped $CH_4$ in System 4 was only occasionally detected. The lack of trapped $CH_4$ seems to indicate that methanogenesis was inhibited by the high concentration of $SO_4^{2-}$ in the CT of System 4.

As shown in Figure C-16, the pH of the cap water and of the CT porewater of System 4 was mostly in the range of 7.8 to 8.2. As discussed in Section 6.2.1, in this slightly alkaline pH range, the major form of the sulphide will be $HS^-$ and the predominant sinks for sulphide will be as the minerals of FeS and $FeS_2$. Gaseous $H_2S$ will not be a dominant sulphur species at these pH values. The trapped gas analysis (see Table B-12) only shows occasional $H_2S$ detection (usually < 1 vol%) in System 4, supporting the above discussion.

Table B-8 shows the $S^{2-}$ analysis results by CHEMetrics test kit on the cap water and the CT porewater of System 4 over 1 y of incubation. In comparison to the MFT in System 3, sulphide was detected more often in both the cap water and CT porewater of System 4. This sulphide most likely results from $SO_4^{2-}$ reduction by SRB. The CT in System 4 had originally a high $SO_4^{2-}$ concentration and thus will favour high SRB activity. However, the sulphide concentration in System 4 was still low, less than 1 mg/L for the most part.

The cap water of System 4 had a higher $SO_4^{2-}$ concentration than the CT porewater over time. This higher $SO_4^{2-}$ concentration in the cap water may be due to the initially rapid release of CT porewater at the beginning of incubation. This rapidly released CT water would not have been subject to as much SRB activity due to the insufficient time for SRB to reduce $SO_4^{2-}$ before its release. The constantly high $SO_4^{2-}$ concentration in the cap water of System 4 over 1 y of incubation may also suggest that $SO_4^{2-}$ reduction by SRB in the cap water did not exist. The cap water had a redox potential of around +400 mV. This high Eh will inhibit the SRB activity.

6.3.2.4 Solids content

Figure C-20 shows the solids content in the CT of System 4 over 1 y of incubation. The baseline solids content in the CT for System 4 (Base CT 3) was 56.6 wt% (see Table B-6).

Figure C-20 shows that the solids content in the CT increased most rapidly in the first 60 d of incubation, and reached 69.3 wt% in CT-U and 70.9 wt% in CT-L at 61 d of incubation. After that time, the CT showed a slow increase in solids content over the remainder of the incubation, and reached 75.9 wt% in CT-U and 76.4 wt% in CT-L at 365 d of incubation. Over 65% of the solids content increase in the CT during 1 y of incubation occurred in the first 61 d of incubation. The CT solids content increase (see Figure C-20) was well matched with the CT tailings volume decrease (see Figure B-6). Both the solids

content increase and the tailings column change indicate rapid CT settling in the first 60 d of incubation and continuous but slow densification over the remainder of incubation time. Furthermore, Figure C-20 shows that the lower zone of CT had a slightly higher solids content than the upper zone of CT, indicating slightly higher densification rate in CT-L than in CT-U.

MacKinnon *et al.* (2000) state that it would take days or weeks for the CT deposit to densify from a solids content of 60 wt% up to 75 wt%. In this project, the CT solids content in System 4 increased from 56.6 wt% to approximately 76.0 wt% after 365 d of incubation.

### *6.3.3 Physical and chemical characteristics in System 1 (Cap water/MFT/CT)*

The CT beneath MFT deposit is a complex system. Many physical, chemical, and microbiological reactions and interactions will occur among the multi-phases of water, MFT, and CT.

In this CT beneath MFT system, the water released from the CT will make its way upward through the overlying MFT. As this CT water passes through the overlying MFT layer, several effects will take place, including simple mixing between CT porewater and MFT porewater for both conservative and reactive components, chemical reactions (e.g. ion exchange, precipitation, and dissolution) and biochemical activities (e.g. sulphate reduction by SRB) for such components as $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, $HCO_3^-$, pH, and $Na^+$. The anaerobic

microbial characteristics of the overlying MFT will also be changed due to the high $SO_4^{2-}$ concentration from the CT release water.

6.3.3.1 Bromide tracer analysis and the estimation of dilution effect

The use of $Br^-$ as a tracer in the CT of System 1 is expected to reveal information about the transit characteristics of CT release water through the MFT and whether the CT water is mixing or displacing the MFT water in the MFT layer, and to estimate the dilution effect at reducing the levels of major water components of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and EC in the cap water. As in System 4, approximately 0.22 g of NaBr was added to 1 L CT at column filling for System 1 (see Table 4-3). As provided in Section 6.3.2.1, this $Br^-$ dosage will produce an expected baseline $Br^-$ concentration of 258 mg/L.

In the MFT of System 1, the baseline $Br^-$ concentration was below detection limits (<0.5 mg/L, in Base MFT 1), the same baseline $Br^-$ concentration as in the MFT of System 3. In the CT of System 1, the baseline $Br^-$ concentration was 270 mg/L for Base CT 1 and 260 mg/L for Base CT 2 (see Table B-6), similar to 260 mg/L of the baseline $Br^-$ concentration in the CT of System 4. Figure C-21 shows the $Br^-$ concentration in the cap water, MFT, and CT over 1 y of incubation in System 1.

In the MFT, the $Br^-$ concentration increased steadily from a non-detectable concentration at incubation time 0 to 130 mg/L in MFT-U and 210 mg/L in MFT-L at 62 d of incubation. Following this initial increase, the $Br^-$

concentration in the MFT layer remained quite constant and in the range of 120 to 150 mg/L in MFT-U zone and of 160 to 210 mg/L in MFT-L zone. The consistently higher $Br^-$ concentration in the MFT-L zone than in the MFT-U zone is possibly due to the direct contact of the MFT-L zone with the high $Br^-$ concentration CT layer.

In the CT, it can be seen from Figure C-21 that the $Br^-$ concentration dropped from baseline 270 mg/L (Table B-6) to 210 mg/L in the first 3 d of incubation and then increased steadily to 270 mg/L at 29 d of incubation. Following this, the $Br^-$ concentration in the CT was quite constant and in the range of 220 to 280 mg/L. This $Br^-$ concentration range is close to the baseline $Br^-$ concentrations of 260 mg/L (Base CT 2) or 270 mg/L (Base CT 1) in CT, indicating $Br^-$ is a stable tracer in the CT material, as in System 4. The $Br^-$ concentration in CT-U was slightly lower than in CT-L, possibly due to the direct contact of CT-U zone with the low $Br^-$ concentration MFT layer.

In the cap water, the $Br^-$ concentration increased quickly to 82 mg/L in the first 29 d of incubation, and then showed a slow but steady increase to a concentration in the range of 70 to 110 mg/L over the remaining incubation.

As in System 4, a mass balance of $Br^-$ in the whole column of System 1 was attempted. System 1 contains MFT and CT at time 0, and cap water, MFT, and CT during incubation. The $Br^-$ concentrations in the cap water, and MFT and CT porewaters were measured for each sacrificed column in System 1 (Table B-9). The cap water volume in each sacrificed column of System 1 can be calculated by using the absolute height of cap water at column sacrificing

(see Table B-3). It should be noted that the column ID is 12.1 cm. For the porewater content, Table C-2 provides the empirical estimations of the masses of solids and porewater per unit volume of MFT and CT at specific solids content.

Then using the same procedures as for the mass balance calculation of $Br^-$ in System 4 (see Section 6.3.2.1), Table 6-10 summarises the $Br^-$ mass calculations in the baseline MFT (Base MFT 1), in the baseline CT (Base CT 1 and Base CT 2), in the cap water, and in the MFT and CT porewaters for the sacrificed columns of System 1. It is seen that over 1 y of incubation, the $Br^-$ recovery (relative to the $Br^-$ mass at time 0) in System 1 was for the most part in the range of 92 to 108%. This indicates that the $Br^-$ is a stable tracer with almost 100% recovery in the MFT and CT materials over incubation time.

**Table 6-10. The $Br^-$ mass balance calculation in System 1.**

| Column | $t_{incu}$ (d) | Mass of $Br^-$ at time 0 (mg) | | | Mass of $Br^-$ at column sacrificing (mg) | | | | | | $Br^-$ mass recovery (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | In MFT | In CT | Total mass of $Br^-$ in the whole column | In cap water | In MFT-U | In MFT-L | In CT-U | In CT-L | Total mass of $Br^-$ in the whole column | |
| C1-2 | 3 | 0 | 1124 | 1124 | 11 | 29 | 96 | 384 | 369 | 889 | 79 |
| C1-3 | 15 | 0 | 1120 | 1120 | 64 | 64 | 153 | 366 | 304 | 951 | 85 |
| C1-4 | 29 | 0 | 1115 | 1115 | 120 | 105 | 204 | 369 | 323 | 1121 | 101 |
| C1-5 | 62 | 0 | 1115 | 1115 | 121 | 171 | 275 | 292 | 307 | 1166 | 105 |
| C1-6 | 91 | 0 | 1124 | 1124 | 140 | 149 | 248 | 300 | 313 | 1150 | 102 |
| C1-12 | 121 | 0 | 1082 | 1082 | 188 | 170 | 231 | 264 | 268 | 1121 | 104 |
| C1-8 | 153 | 0 | 1111 | 1111 | NA | 159 | 245 | 255 | 272 | NA | NA |
| C1-9 | 182 | 0 | 1124 | 1124 | 182 | 150 | 219 | 255 | 276 | 1082 | 96 |
| C1-10 | 213 | 0 | 1122 | 1122 | 223 | 191 | 270 | 260 | 256 | 1200 | 107 |
| C1-11 | 244 | 0 | 1132 | 1132 | 225 | 175 | 238 | 238 | 282 | 1158 | 102 |
| C1-13 | 267 | 0 | 1086 | 1086 | 245 | 185 | 234 | 251 | 261 | 1176 | 108 |
| C1-14 | 303 | 0 | 1090 | 1090 | 281 | NA | NA | NA | NA | NA | NA |
| C1-15 | 335 | 0 | 1090 | 1090 | 235 | 162 | 184 | 206 | 220 | 1007 | 92 |
| C1-16 | 366 | 0 | 1096 | 1096 | 258 | 186 | 222 | 246 | 233 | 1145 | 104 |

1). $Br^-$ mass recovery (%) =((total mass of $Br^-$ at column sacrificing)/(total mass of $Br^-$ at time 0))×100%.
2). NA=not available.

The MFT originally contained no detectable $Br^-$. The detected concentration of $Br^-$ in the MFT during incubation is the result of $Br^-$ entering the MFT from the underlying CT release water. The concentration drop in $Br^-$ in the MFT layer relative to the $Br^-$ concentration in the CT layer is most likely due to the dilution effect between the MFT water and CT water.

As indicated by the small increase in MFT solids content and the small decrease in MFT tailings volume over time, the MFT in System 1 densified slowly over time. The water content in MFT can therefore be roughly regarded as unchanged over a short time period. Thus, in the MFT layer of System 1, the sum of MFT water and CT release water is a relatively constant amount. As the CT water moves upward through the MFT into the cap water, a fraction of the CT release water will replace the MFT water in the MFT layer and move up together into the cap water layer. Therefore, during the incubation, the water in the MFT layer and the cap water layer contains a mixture of CT water and MFT water.

The volume percents of MFT and CT water (vol%) in the MFT and cap water layers can be calculated simply from the $Br^-$ concentration, by assuming that the $Br^-$ is a conservative species. Table 6-11 shows the percents (vol%) of CT water in the cap water and MFT layers in System 1 over 1 y of incubation, which are calculated by dividing the $Br^-$ concentrations in the cap water and MFT layers by the $Br^-$ concentration in the baseline CT. For example, for cap water at 3 d of incubation, the $Br^-$ concentration is 25 mg/L (see Table B-9), so the CT water percent in this cap water is (25/270)*100=9 vol%. The balance

water in Table 6-11 is the baseline MFT water, in which the $Br^-$ is below detection limits (<0.5 mg/L).

**Table 6-11. The volume percent (vol%) of CT water with balance MFT water, based on $Br^-$ analysis in System 1.**

| $t_{incu}$ (d) | Percent of CT water in cap water and MFT layers (vol%) | | |
|---|---|---|---|
| | Cap water | MFT-U | MFT-L |
| 3 | 9 | 9 | 29 |
| 15 | 24 | 19 | 44 |
| 29 | 32 | 30 | 59 |
| 62 | 26 | 48 | 78 |
| 91 | 28 | 44 | 74 |
| 121 | 34 | 52 | 70 |
| 153 | NA | 48 | 74 |
| 182 | 36 | 47 | NA |
| 213 | 41 | 56 | 78 |
| 244 | 41 | 52 | 70 |
| 267 | 41 | 56 | 70 |
| 303 | 44 | 56 | 67 |
| 335 | 41 | 52 | 59 |
| 366 | 41 | 56 | 67 |

1). Use 270 mg/L as the baseline $Br^-$ concentration in the CT (see Table B-6),
2). The baseline $Br^-$ concentration in the MFT is assumed to be 0 mg/L,
3). NA=not available.

Table 6-11 shows that, as the CT water flows upward to the cap water, the CT water will replace the MFT water in MFT layer and move up together with the MFT water into the cap water. For example, at 366 d of incubation, the CT water replaced 67 vol% of MFT-L porewater, 56 vol% of MFT-U porewater; and the cap water contained 41 vol% of CT water with remaining 59 wt% of MFT water.

The $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and electrical conductivity were the major water parameters considered in this study. In System 1, the baseline CT had higher concentrations of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and higher EC than the baseline MFT (see

Table B-6). Thus, the mixing of MFT and CT water in the cap water will reduce the concentrations of these species from the concentrations in the CT simply by dilution effect. Therefore, in the cap water of System 1, the decrease in $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and EC relative to the levels in baseline CT might be caused by both simple dilution and other removal mechanisms from the water phase (such as precipitation and microbial reduction).

The dilution effect ($P_d$) is defined in Equation 6-11:

$$P_d\ (\%) = ((C_{Base\ CT} - C_d)/(C_{Base\ CT} - C_m)) \times 100\% \quad (6\text{-}11)$$

where $C_d$ is the ion concentration or EC in the cap water calculated by simple dilution between CT and MFT waters; $C_m$ is the measured ion concentration or EC in the cap water; and $C_{Base\ CT}$ is the baseline ion concentration or EC in the CT.

From the $P_d$% definition in Equation 6-11, increased $P_d$% value means increased dilution effect. Based on the percent (vol%) of MFT and CT waters (see Table 6-11), and the $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ concentrations and EC in baseline MFT, in baseline CT, and in the cap water during incubation (see Tables B-6 and B-9), the dilution effect ($P_d$) at reducing the $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ concentrations and the EC for the cap water can be estimated, as shown in Table 6-12.

**Table 6-12. The estimation of dilution effect ($P_d$) at reducing $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ concentrations, and the EC for the cap water of System 1.**

| | Makeup of the cap water | | $Ca^{2+}$ in the cap water | | $Mg^{2+}$ in the cap water | | $SO_4^{2-}$ in the cap water | | EC in the cap water | |
|---|---|---|---|---|---|---|---|---|---|---|
| $t_{incu}$ (d) | Vol% of CT water | Vol% of MFT water | $C_d$ (mg/L) | $P_d$ (%) | $C_d$ (mg/L) | $P_d$ (%) | $C_d$ (mg/L) | $P_d$ (%) | $C_d$ (μS/cm) | $P_d$ (%) |
| 3 | 9 | 91 | 26.7 | 94 | 16.4 | 107 | 67 | 108 | 4554. | 105 |
| 15 | 24 | 76 | 33.7 | 84 | 17.8 | 96 | 177 | 112 | 4757 | 110 |
| 29 | 32 | 68 | 37.2 | 77 | 18.5 | 86 | 231 | 100 | 4857 | 96 |
| 62 | 26 | 74 | 34.4 | 80 | 17.9 | 91 | 188 | 82 | 4777 | 97 |
| 91 | 28 | 72 | 35.5 | 74 | 18.1 | 78 | 206 | 72 | 4811 | 79 |
| 121 | 34 | 66 | 40.7 | 68 | 19.0 | 70 | 306 | 73 | 5006 | 89 |
| 182 | 36 | 64 | 39.0 | 70 | 18.8 | 80 | 260 | 71 | 4911 | 98 |
| 213 | 41 | 59 | 41.2 | 63 | 19.3 | 73 | 295 | 64 | 4975 | 88 |
| 244 | 41 | 59 | 41.2 | 63 | 19.3 | 73 | 295 | 73 | 4975 | 98 |
| 267 | 41 | 59 | 44.2 | 60 | 20.0 | 56 | 365 | 75 | 5118 | 90 |
| 303 | 44 | 56 | 46.2 | 65 | 20.0 | 78 | 399 | 80 | 5181 | 110 |
| 335 | 41 | 59 | 44.2 | 68 | 20.0 | 90 | 365 | 81 | 5118 | 98 |
| 366 | 41 | 59 | 44.2 | 65 | 20.0 | 70 | 365 | 85 | 5118 | 103 |

1). The symbols used in Table 6-12 are explained in Equation 6-11.

2). For MFT, the baseline data is from "Base MFT 1" (see Table B-6); For CT, the baseline data is from "Base CT 1" or "Base CT 2" (see Table B-6).

3). Sample calculation for $Ca^{2+}$ at 3 d of incubation:

a). The makeup of this cap water is 9 vol% of CT water and 91 vol% of MFT water (see Table 6-11).

b). The baseline $Ca^{2+}$ concentration was 68.7 mg/L for CT (Base CT 1) and 22.4 mg/L for MFT (Base MFT 1) (see Table B-6).

c). The $C_d$ of $Ca^{2+}$ concentration in the cap water achieved by simple dilution =((68.7×9) + (22.4×91))/100 =26.6 mg/L.

d). The $C_m$ of measured $Ca^{2+}$ concentration in the cap water at 3 d of incubation was 24 mg/L.

e). The dilution effect ($P_d$ %) at reducing the $Ca^{2+}$ concentration for the cap water = ((68.7-26.6)/(68.7-24))*100%=94%.

The estimation of $P_d$ will be severely affected if there are any dissolutions or inputs of these water parameters (i.e. $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ and EC) from the MFT and CT tailings into the porewater during the incubation period. Despite the possible sources of the estimation error, the $P_d$ at Table 6-12 can still show that dilution played a large role at reducing the $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ concentrations and EC for the cap water in System 1. This result indicates that, in the co-disposal of CT beneath MFT system (System 1), a large volume ratio of MFT to CT will be favoured for the purpose of increasing the dilution effects at reducing the $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ concentrations and the EC for the cap water.

6.3.3.2 Calcium and magnesium concentrations

Figure C-22 shows $Ca^{2+}$ concentration in System 1 over 1 y of incubation.

From Table B-6, the baseline $Ca^{2+}$ concentration in the MFT for System 1 (Base MFT 1) was 22 mg/L, similar to 23 mg/L of baseline $Ca^{2+}$ concentration in the MFT for System 3 (Base MFT 2). In the MFT layer of System 1, Figure C-22 shows that the $Ca^{2+}$ concentration decreased slightly and mainly at the beginning of incubation. At 15 d of incubation, the $Ca^{2+}$ concentration in the MFT layer decreased to 17 mg/L in MFT-U and 19 mg/L in MFT-L. After that time, the $Ca^{2+}$ concentration decreased slowly to 13 mg/L in MFT-U and 14 mg/L in MFT-L at 366 d of incubation. The upper zone of MFT had a slightly lower $Ca^{2+}$ concentration than the lower zone of MFT. This slight difference in $Ca^{2+}$ concentration is likely due to the fact that the MFT-L zone was in direct

contact with the CT layer, which had initially high $Ca^{2+}$ concentration. The MFT had consistently lower $Ca^{2+}$ concentration than the CT in System 1 over 1 y of incubation. The $Ca^{2+}$ concentration in the MFT of System 1 was similar to the $Ca^{2+}$ concentration in the MFT of System 3 (see Figure C-8) over 1 y of incubation.

From Table B-6, the baseline $Ca^{2+}$ concentration in the CT was 69 mg/L for columns C1-1 to C1-11 (Base CT 1) and 76 mg/L for columns C1-12 to C1-20 (Base CT 2) in System 1. In comparison, the baseline $Ca^{2+}$ concentration in the CT for System 4 (Base CT 3) was 82 mg/L (see Table B-6). In the CT layer, Figure C-22 shows that the largest drop in $Ca^{2+}$ concentration occurred during the first 30 d of incubation. By 29 d of incubation, the $Ca^{2+}$ concentration in the CT decreased substantially to 29 mg/L in CT-U and 23 mg/L in CT-L. After that time, the $Ca^{2+}$ concentration remained quite constant with 21 mg/L in CT-U and 18 mg/L in CT-L at 366 d of incubation. The upper and lower zones of CT had similar $Ca^{2+}$ concentrations over 1 y of incubation. The $Ca^{2+}$ concentration in the CT of System 1 was similar to the $Ca^{2+}$ concentration in the CT of System 4 (see Figure C-15) during 1 y of incubation.

In the cap water, Figure C-22 indicates that the $Ca^{2+}$ concentration was quite constant with time. During 1 y of incubation, the $Ca^{2+}$ concentration in the cap water was in the range of 23 and 30 mg/L. From Figure C-22, it can be seen that the cap water had a $Ca^{2+}$ concentration close to that in the CT and higher than that in the MFT. This $Ca^{2+}$ concentration difference may be partially due to the pH difference between the cap water and MFT porewater. Compared to

MFT porewater, the cap water had a lower pH over 1 y of incubation (see Figure C-23). This lower pH may contribute to the slightly higher $Ca^{2+}$ concentration in the cap water due to the possible redissolution of $CaCO_3$ under lowered pH conditions.

Using the same procedures as for the $Br^-$ mass balance calculations in Section 6.3.3.1, a mass balance of $Ca^{2+}$ in the whole column of System 1 was attempted. Table 6-13 lists the results of $Ca^{2+}$ mass balance calculations in System 1.

It can be seen from Table 6-13 that, over 1 y of incubation, only less than 50% of the original $Ca^{2+}$ mass still exits in the water, suggesting that more than 50% of the original $Ca^{2+}$ mass was removed from the water by some mechanisms. System 1 showed similar degree of overall $Ca^{2+}$ removal from the water as in System 4 (Table 6-7).

The mechanisms causing $Ca^{2+}$ concentration changes in the MFT and CT of System 1 may include mixing and dilution of high $Ca^{2+}$ concentration CT porewater by low $Ca^{2+}$ concentration MFT porewater, precipitation as $CaCO_3$ at slightly alkaline conditions or redissolution of $CaCO_3$ at lowered pH conditions, and ion exchange between $Ca^{2+}$ and $Na^+$ on the clays of MFT and CT. The dilution effect in reducing $Ca^{2+}$ concentration is already discussed in Section 6.3.3.1.

**Table 6-13. The $Ca^{2+}$ mass balance calculation in System 1.**

| Column | $t_{incu}$ (d) | Mass of $Ca^{2+}$ at time 0 (mg) | | | Mass of $Ca^{2+}$ at column sacrificing (mg) | | | | | | $Ca^{2+}$ mass recovery (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | In MFT | In CT | Total mass of $Ca^{2+}$ in the whole column | In cap water | In MFT-U | In MFT-L | In CT-U | In CT-L | Total mass of $Ca^{2+}$ in the whole column | |
| C1-2 | 3 | 57 | 286 | 343 | 10 | 25 | 26 | 93 | 89 | 243 | 71 |
| C1-3 | 15 | 56 | 285 | 341 | 26 | 21 | 24 | 55 | 55 | 181 | 54 |
| C1-4 | 29 | 57 | 284 | 341 | 39 | 22 | 24 | 40 | 28 | 153 | 45 |
| C1-5 | 62 | 57 | 284 | 341 | 44 | 22 | 23 | 32 | 30 | 151 | 45 |
| C1-6 | 91 | 56 | 286 | 342 | 44 | 21 | 21 | 26 | 29 | 141 | 41 |
| C1-12 | 121 | 56 | 316 | 372 | 50 | 21 | 25 | 35 | 32 | 163 | 43 |
| C1-8 | 153 | 56 | 283 | 339 | NA | 18 | 21 | 24 | 23 | NA | NA |
| C1-9 | 182 | 56 | 286 | 342 | 49 | 20 | 24 | 28 | 27 | 148 | 43 |
| C1-10 | 213 | 57 | 285 | 342 | 51 | 15 | 20 | 26 | 27 | 139 | 41 |
| C1-11 | 244 | 57 | 288 | 345 | 51 | 17 | 19 | 28 | 32 | 147 | 42 |
| C1-13 | 267 | 57 | 301 | 358 | 51 | 15 | 18 | 23 | 25 | 132 | 37 |
| C1-14 | 303 | 57 | 302 | 359 | 71 | NA | NA | NA | NA | NA | NA |
| C1-15 | 335 | 57 | 302 | 359 | 63 | 17 | 18 | 23 | 26 | 147 | 41 |
| C1-16 | 366 | 57 | 304 | 361 | 63 | 16 | 18 | 21 | 18 | 136 | 38 |

1). $Ca^{2+}$ mass recovery (%) =((total mass of $Ca^{2+}$ at column sacrificing)/(total mass of $Ca^{2+}$ at time 0))×100%.

2). NA=not available.

The precipitation or redissolution of $CaCO_3$ was discussed previously in Section 6.3.2.2 and Equation 6-8. Figure C-23 shows that the pH in the cap water and MFT and CT porewaters of System 1 were mostly between 7.6 and 8.3, showing slightly alkaline conditions. Due to the high $Ca^{2+}$ concentration and slightly alkaline pH in the CT porewater, the reduction in $Ca^{2+}$ concentration in the CT of System 1 in the first 29 d of incubation was likely due to this $CaCO_3$ precipitation.

Using the same methods as for the calculation of the ratio of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in System 4 (Section 6.3.2.2), Table 6-14 summarises the ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in each zone of System 1 over 1 y of incubation. Table 6-14 shows that the ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in all zones of System 1 over 1 y of incubation are in the range of 3 to 12, indicating that the precipitation of $CaCO_3$ is a possible route for the removal of $Ca^{2+}$ in System 1.

**Table 6-14. The ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in the cap water and porewaters of System 1 at column sacrificing.**

| Column | $t_{locu}$ (d) | The ratio of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp | | | | |
|---|---|---|---|---|---|---|
| | | In cap water | In MFT-U | In MFT-L | In CT-U | In CT-L |
| C1-2 | 3 | 9 | | 4 | 11 | 12 |
| C1-3 | 15 | 5 | 5 | 5 | 9 | 12 |
| C1-4 | 29 | 7 | 5 | 6 | 14 | 12 |
| C1-5 | 62 | NA | 6 | 10 | 12 | 6 |
| C1-6 | 91 | 5 | 7 | 7 | 7 | 9 |
| C1-12 | 121 | 4 | 4 | 7 | 7 | 7 |
| C1-8 | 153 | | 3 | 4 | 4 | 6 |
| C1-9 | 182 | 4 | 5 | 8 | 7 | 9 |
| C1-10 | 213 | 4 | 5 | 7 | 5 | 7 |
| C1-11 | 244 | 3 | 3 | 5 | 5 | 10 |
| C1-13 | 267 | 3 | 3 | 4 | 3 | 5 |
| C1-14 | 303 | NA | NA | NA | NA | NA |
| C1-15 | 335 | 5 | 5 | 5 | 15 | 14 |
| C1-16 | 366 | 10 | 4 | 4 | 10 | 5 |

Ion exchange between $Ca^{2+}$ and $Na^{+}$ on the clays of the MFT and CT is another important mechanism for $Ca^{2+}$ concentration reduction in MFT and CT, as expressed in the following equation:

$$Ca^{2+} + 2\text{Na-clay} \rightleftharpoons 2Na^{+} + \text{Ca-clay} \quad \text{.....(6-12)}$$

Thus, the drop in $Ca^{2+}$ concentration caused by ion exchange between $Ca^{2+}$ and $Na^{+}$ will result in an increase in the $Na^{+}$ concentration in the porewater. Figure C-24 shows the $Na^{+}$ concentration in System 1 over time. From Figure C-24, it can be seen that the $Na^{+}$ concentrations in the cap water and MFT and CT

porewaters increased over time. The increase in $Na^+$ concentration is a possible indicator of ion exchange between $Ca^{2+}$ and $Na^+$.

Figure C-25 shows the $Mg^{2+}$ concentration in System 1 over 1 y of incubation. Similar to $Ca^{2+}$, in System 1, the CT had baseline $Mg^{2+}$ concentration of 25 mg/L for Base CT 1 and 26 mg/L for Base CT 2, higher than 16 mg/L of baseline $Mg^{2+}$ concentration for MFT. In the MFT, the $Mg^{2+}$ concentration was quite constant and in the range of 11 to 16 mg/L over 1 y of incubation. In the CT, the major drop in $Mg^{2+}$ concentration occurred in the 30 d of incubation and reached 14 mg/L in CT-U and 15 mg/L in CT-L at 29 d of incubation. After that time, the $Mg^{2+}$ concentration in the CT was quite constant. In the cap water, the $Mg^{2+}$ concentration was in the range of 16 to 19 mg/L, higher than the concentration in either MFT or CT.

6.3.3.3 Sulphate concentration

Figure C-4 shows the $SO_4^{2-}$ concentration in System 1 over 1 y of incubation.

From Table B-6, in the MFT of System 1, the baseline $SO_4^{2-}$ concentration in the MFT (Base MFT 1) was below detection limits (<0.5 mg/L). In comparison, the baseline $SO_4^{2-}$ concentration in the MFT for System 3 (Base MFT 2) was 14 mg/L. In the MFT, Figure C-4 shows that the $SO_4^{2-}$ concentration increased rapidly at the beginning of incubation and reached 115 mg/L in MFT-U and 226 mg/L in MFT-L at 3 d of incubation. This rapid increase in $SO_4^{2-}$ concentration in the MFT at the early incubation times was

most likely due to the rapid release of the underlying CT water. Following this initial increase, the $SO_4^{2-}$ concentration in the MFT decreased. Most of the $SO_4^{2-}$ concentration drop occurred in the first 30 d of incubation, with the $SO_4^{2-}$ concentration reaching 31 mg/L in MFT-U and 87 mg/L in MFT-L at 29 d of incubation. After that time, the $SO_4^{2-}$ concentration remained relatively constant with 44 mg/L in MFT-U and 60 mg/L in MFT-L at 366 d of incubation. The lower zone of MFT had higher $SO_4^{2-}$ concentration than the upper zone of MFT, likely due to the fact that the MFT-L zone was in direct contact with the underlying CT layer (which had a high $SO_4^{2-}$ concentration).

From Table B-6, it can be seen that the baseline $SO_4^{2-}$ concentration in the CT was 724 mg/L for columns C1-1 to C1-11 (Base CT 1) and 897 mg/L for C1-12 to C1-20 (Base CT 2). In comparison, the baseline $SO_4^{2-}$ concentration in the CT for System 4 (Base CT 3) was 1100 mg/L. In the CT of System 1, Figure C-4 indicates that the largest drop in $SO_4^{2-}$ concentration occurred during the first 30 d of incubation. At 29 d of incubation, the $SO_4^{2-}$ concentration in the CT decreased substantially to 336 mg/L in CT-U and 260 mg/L in CT-L. After that time, the $SO_4^{2-}$ concentration decreased and reached 21 mg/L in CT-U and 23 mg/L in CT-L at 366 d of incubation, corresponding to more than 95% $SO_4^{2-}$ concentration reduction. With the exception of a few data points, the two CT-U and CT-L zones showed similar $SO_4^{2-}$ concentrations over time. Furthermore, the CT had consistently higher $SO_4^{2-}$ concentration than the MFT over 1 y of incubation. At the end of 1 y of incubation, however, both the CT and MFT in System 1 approached similar $SO_4^{2-}$ concentrations in the range of 21 to 60 mg/L.

In the cap water, Figure C-4 shows that the $SO_4^{2-}$ concentration increased rapidly at the beginning of the incubation and reached a maximum value of 235 mg/L at 15 d of incubation. This rapid increase in $SO_4^{2-}$ concentration in cap water at the early incubation was likely from the rapidly released CT water. Following this initial increase, the $SO_4^{2-}$ concentration decreased, and dropped to 73 mg/L at 62 d of incubation. After that time, the $SO_4^{2-}$ concentration remained stable until 213 d of incubation with a $SO_4^{2-}$ concentration of 53 mg/L. From 244 d of incubation, the $SO_4^{2-}$ concentration in the cap water increased with time and reached 277 mg/L at 304 d of incubation and 271 mg/L at 366 d of incubation. These $SO_4^{2-}$ concentrations in the cap water were even higher than in either MFT or CT layer. The reasons causing this increase in $SO_4^{2-}$ concentration in the cap water were unknown.

Using the same procedures as for the $Br^-$ mass balance calculations in Section 6.3.3.1, a mass balance of $SO_4^{2-}$ in the whole column of System 1 was attempted. Table 6-15 lists the results of $SO_4^{2-}$ mass balance calculations in System 1.

**Table 6-15. The $SO_4^{2-}$ mass balance calculation in System 1.**

| Column | $t_{incu}$ (d) | Mass of $SO_4^{2-}$ at time 0 (mg) | | | Mass of $SO_4^{2-}$ at column sacrificing (mg) | | | | | | $SO_4^{2-}$ mass recovery (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | In MFT | In CT | Total mass of $SO_4^{2-}$ in whole column | In cap water | In MFT-U | In MFT-L | In CT-U | In CT-L | Total mass of $SO_4^{2-}$ in the whole column | |
| C1-2 | 3 | 0 | 3013 | 3013 | 50 | 159 | 282 | 1248 | 1213 | 2952 | 98 |
| C1-3 | 15 | 0 | 3002 | 3002 | 230 | 65 | 173 | 749 | 636 | 1853 | 62 |
| C1-4 | 29 | 0 | 2991 | 2991 | 318 | 40 | 111 | 459 | 311 | 1239 | 41 |
| C1-5 | 62 | 0 | 2991 | 2991 | 126 | 39 | 139 | 321 | 323 | 948 | 32 |
| C1-6 | 91 | 0 | 3013 | 3013 | 0 | 21 | 78 | 204 | 225 | 528 | 18 |
| C1-12 | 121 | 0 | 3734 | 3734 | 185 | 50 | 115 | 279 | 75 | 704 | 19 |
| C1-8 | 153 | 0 | 2980 | 2980 | NA | 15 | 73 | 154 | 154 | NA | NA |
| C1-9 | 182 | 0 | 3013 | 3013 | 128 | 27 | 64 | 73 | 36 | 328 | 11 |
| C1-10 | 213 | 0 | 3008 | 3008 | 106 | 24 | 79 | 226 | 253 | 688 | 23 |
| C1-11 | 244 | 0 | 3035 | 3035 | 272 | 41 | 73 | 119 | 99 | 604 | 20 |
| C1-13 | 267 | 0 | 3748 | 3748 | 419 | 55 | 81 | 111 | 94 | 760 | 20 |
| C1-14 | 303 | 0 | 3762 | 3762 | 649 | NA | NA | NA | NA | NA | NA |
| C1-15 | 335 | 0 | 3762 | 3762 | 507 | 26 | 47 | 50 | 99 | 729 | 19 |
| C1-16 | 366 | 0 | 3782 | 3782 | 635 | 54 | 74 | 22 | 23 | 808 | 21 |

1). $SO_4^{2-}$ mass recovery (%) =((total mass of $SO_4^{2-}$ at column sacrificing)/(total mass of $SO_4^{2-}$ at time 0))×100%.
2). NA=not available.

It is seen from Table 6-15 that, after 1 y of incubation, only less than 20% of the original $SO_4^{2-}$ mass still exits in the water, suggesting that more than 80% of the original $SO_4^{2-}$ mass was removed from the water by some mechanisms in System 1. System 1 removed more $SO_4^{2-}$ than System 4 over same incubation periods.

As discussed previously in Section 2.4, with its high $SO_4^{2-}$ concentration, the conditions in the CT should favour the sulphate reduction by SRB when the environment becomes anaerobic. The MFT in System 1 will receive CT release water, which may also result in a high enough $SO_4^{2-}$ concentration in the MFT to stimulate the SRB activity. The following discussions about the results of $HCO_3^-$, Eh, and trapped gas analysis are used to determine whether sulphate reduction by SRB was occurring in the CT and MFT of System 1.

As shown in Equation 2-2, the sulphate reduction by SRB will produce $HCO_3^-$. Figure C-26 shows the $HCO_3^-$ concentration in System 1.

In the CT of System 1, the baseline $HCO_3^-$ concentration for CT was 739 mg/L in Base CT 1 and 806 mg/L in Base CT 2 (see Table B-6). Figure C-26 shows a steady increase in $HCO_3^-$ concentration in the CT over time. At 366 d of incubation, the $HCO_3^-$ concentration reached 1350 mg/L in CT-U and 1310 mg/L CT-L, corresponding to approximately a 65% increase from the baseline data. The increase in $HCO_3^-$ concentration in the CT of System 1 was well matched with the decrease in $SO_4^{2-}$ concentration (see Figure C-4) over 1 y of incubation, indicating the SRB reduction of $SO_4^{2-}$ in the CT layer.

In the MFT of System 1, the baseline $HCO_3^-$ concentration for MFT (Base MFT 1) was 1491 mg/L. As shown in Figure C-26, at the beginning of the incubation, the MFT experienced a slight drop in $HCO_3^-$ concentration, possibly due to the mixing and dilution with the low $HCO_3^-$ concentration upflowing CT water. After 15 d of incubation, the $HCO_3^-$ concentration in the MFT increased slowly until 182 d of incubation. The slight increase in $HCO_3^-$ concentration of MFT was matched with the slight decrease in $SO_4^{2-}$ concentration of MFT (see Figure C-4), implying the activity of sulphate reduction by SRB in the MFT layer. From 210 to 365 d of incubation, the $HCO_3^-$ concentration in MFT dropped slightly. This may be caused by some unknown factors.

The redox potential may also provide an indicator for favourable conditions for $SO_4^{2-}$ reduction by SRB. Figure C-27 shows the Eh in the MFT and CT of System 1 over 1 y of incubation. It can be seen that, after the first 3 to 15 d of incubation, the Eh in the CT was in the range of –178 to –206 mV, and the Eh in the MFT was for the most part in the range of –110 to –182 mV over 1 y of incubation, indicating anaerobic conditions in both MFT and CT. Most of the Eh values in the MFT and CT of System 1 thus support the presence of anaerobic conditions, which favour the SRB activity.

The trapped gas analysis in System 1 (see Table B-12) also showed the presence of low levels of gaseous $H_2S$ in some samples. The presence of $H_2S$ is likely the result of sulphate reduction by SRB.

6.3.3.4 pH and electrical conductivity

The pH in the cap water and porewaters of MFT and CT in System 1 was mostly in the range of 7.6 to 8.3 (see Figure C-23). The cap water had lower pH than the porewaters of MFT and CT over 1 y of incubation.

Figure C-28 shows the electrical conductivity in System 1 over 1 y of incubation. In System 1, the baseline EC was 4430 μS/cm for MFT (Base MFT 1), 5770 μS/cm for CT in columns C1-1 to C1-11 (Base CT 1), and 6120 μS/cm for CT in columns C1-12 to C1-20 (Base CT 2). The baseline CT had a higher EC than the baseline MFT.

Over 1 y of incubation, the EC in MFT increased and reached 4870 μS/cm in MFT-U and 4940 μS/cm in MFT-L at 366 d of incubation. The MFT-L zone showed slightly higher EC than the MFT-U zone, possibly due to the direct contact of the MFT-L zone with the CT layer, which had higher EC.

The EC in the CT showed a rapid decrease in the first 30 d of incubation. This rapid decrease in EC can be attributed to the rapid decrease in $Ca^{2+}$ (see Figure C-22), $Mg^{2+}$ (see Figure C-25), and $SO_4^{2-}$ (see Figure C-4) concentrations in CT at the beginning of incubation. Following this rapid decrease in EC, the EC in the CT increased slightly with time (except for EC data at 153 d of incubation) and reached 5350 μS/cm in CT-U and 5190 μS/cm in CT-L at 366 d of incubation. Both the upper and lower zones of CT showed similar EC values over time.

In the cap water of System 1, the EC was in the range of 4560 to 5330 μS/cm over 1 y of incubation, and between that of MFT and CT. The EC in the cap water increased slightly over time.

6.3.3.5 The possible sinks of $SO_4^{2-}$ reduction by SRB and the AVS analysis

As discussed above, over 366 d of incubation, both the CT and MFT in System 1 had a $SO_4^{2-}$ concentration greater than 20 mg/L in most analyses. The value of 20 mg/L is believed to be the critical $SO_4^{2-}$ concentration, above which SRB will out-compete methanogens for substrates (Fedorak *et al.* 2002). The Eh in the CT and MFT indicated suitable conditions for the SRB, and the decrease in $SO_4^{2-}$ concentrations and the increase in $HCO_3^-$ concentrations indicate the presence of SRB activity in the MFT and CT of System 1.

In the dissimilatory sulphate reduction, $SO_4^{2-}$ acts as electron acceptor during the oxidation of organic matter by SRB and is reduced to sulphide as shown in Equation 2-2.

The MFT and CT in System 1 had pH in the range of 7.6 to 8.3 (see Figure C-23). In this pH range, the predominant form of sulphide is $HS^-$ rather than $H_2S$ (Fedorak *et al.* 2000). Only a small portion of the sulphide may be recovered as $H_2S$, as shown in Equation 6-13:

$$HS^- + H_2O \rightleftharpoons H_2S_{(g)} + OH^- \quad \text{.....(6-13)}$$

In the presence of heavy metals, such as $Fe^{2+}$, the $HS^-$ will further react with the metals to precipitate as metal sulphides, such as FeS. FeS may be further converted to pyrite, as shown in the following equations:

$$HS^- + Fe^{2+} \longrightarrow FeS_{(s)} + H^+ \quad \text{(6-14)}$$

$$FeS + S^0 \longrightarrow FeS_{2(s)} \quad \text{(6-15)}$$

As noted by Schlesinger (1997), AVS (mainly the iron sulphides) is an effective trap for $H_2S$, which might otherwise escape to the atmosphere. Because AVS compounds are metastable and reactive, either by oxidation or by subsequent reduction to $FeS_2$, it is reasonable to assume that its presence is an indicator of recent $SO_4^{2-}$ reduction (Kennedy *et al.* 1999).

The AVS analysis in this project was performed by the commercial laboratory using 6 M cold HCl and $SnCl_2$ to digest the sample. As discussed in Section 4.2, the analysed sulphur pool was therefore the AVS plus fine-graded (newly formed) synthetic pyrite. The AVS analysis was only conducted on MFT and CT of baseline and after approximately 120 d of incubation, as shown in Table 6-16. Table 6-16 also contains related $SO_4^{2-}$ concentrations (as determined by IC) and $S^{2-}$ concentrations (as determined by CHEMetrics test kit).

**Table 6-16. AVS analysis and related $SO_4^{2-}$ and $S^{2-}$ measurements in System 1.**

| Sample | $t_{incu}$ (d) | AVS (mg/kg of dry sample) | $SO_4^{2-}$ (mg/L) | $S^{2-}$ (mg/L) |
|---|---|---|---|---|
| Base MFT 1 | 0 | 74 | BDL | BDL |
| Base CT 2 | 0 | 80 | 897 | 4.0 |
| C1-12-CapW | 121 | NA | 90 | BDL |
| C1-12-MFT-U | 121 | 150 | 41 | BDL |
| C1-12-MFT-L | 121 | 186 | 94 | BDL |
| C1-12-CT-U | 121 | 29 | 264 | 0.1 |
| C1-12-CT-L | 121 | NA | 68 | 0.1 |

1). NA=not available,
2). BDL=below detection limit.

The AVS results (see Table 6-16) show that, after approximately 120 d of incubation, the AVS increased in the MFT but decreased in the CT from the baseline data. The increase in AVS of the MFT may be attributed to the formation of sulphides as a result of sulphate reduction by SRB. However, the drop in AVS of the CT is quite unreasonable and suspicious. As discussed above, the high $SO_4^{2-}$ concentration, anaerobic environment (as indicated by the low Eh value), and increased $HCO_3^-$ concentration in the CT all support the sulphate reduction by SRB in the CT. The exact reasons for the decrease in AVS of the CT were unable to be identified.

Sulphite ($SO_3^{2-}$) and thiosulphate ($S_2O_3^{2-}$) are possible intermediates in the sulphate reduction to sulphide. In a similar project conducted by Fedorak *et al.* (2000), most of the Syncrude MFT and CT samples showed no detectable $SO_3^{2-}$ concentrations, and only occasional detections of $S_2O_3^{2-}$ with a maximum concentration of approximately 50 mg/L in the CT samples. Therefore, $S_2O_3^{2-}$ is a possible sink of sulphur from $SO_4^{2-}$ reduction.

Another possible sink of sulphur from $SO_4^{2-}$ reduction by SRB is dissolved sulphides in the water. As discussed previously in Section 4.2, the $S^{2-}$ analysis on the cap water and porewater was performed using The CHEMetrics test kit. The CHEMetrics test kit measures the total acid soluble sulphides in water after the water is first filtered through a 0.45 μm filter. Thus, the $S^{2-}$ analysis results will include all the components of dissolved $H_2S$, $HS^-$, and acid soluble metallic sulphides in suspension.

Table B-9 shows the $S^{2-}$ analysis results on the cap water and porewater of MFT and CT in System 1 over 1 y of incubation. It can be seen from Table B-9 that most of the $S^{2-}$ concentrations in the cap water and porewaters of MFT and CT were non-detectable (<0.05 mg/L), with a few samples having low (0.1 to 6.0 mg/L) concentrations of $S^{2-}$. These results indicate that the majority of the sulphide from sulphate reduction by SRB was not in the cap water or porewaters in the form of $S^{2-}$.

6.3.3.6 Solids content

Figure C-29 shows the solids content in the MFT and CT of System 1 over 1 y of incubation. The solids contents of both MFT and CT increased steadily over 1 y of incubation.

In the MFT of System 1, the baseline solids content was 37.9 wt% (Base MFT 1, see Table B-6), similar to 38.0 wt% of the baseline solids content of MFT in System 3. The MFT solids content increased steadily over the entire

year of incubation, and reached 42.5 wt% in MFT-U and 42.9 wt% in MFT-L at 366 d of incubation. This steady increase in the MFT solids content is not consistent with the MFT tailings volume change in System 1 (see Figure C-1). The MFT tailings volume in System 1 did not show a consistent decrease but rather fluctuated over time. The inconsistency between these two data sets can be explained by the following two reasons. First, some measurement error was likely introduced when measuring the Geo-grid position for the calculation of MFT tailings volume change; second, it is possible that gas might be trapped in the MFT, which would affect the data of MFT tailings volume change.

These two factors, however, will not affect the solids content analysis. Therefore, it is assumed that, for the MFT in System 1, the solids content data are more reliable in indicating its consolidation, rather than the data of the MFT tailings volume change.

For the MFT, from its solids content, the released and remaining water percents (vol %) can be empirically estimated. Table C-3 in Appendix C lists the empirical estimation of released and still remaining water contents per litre of original MFT, based on the MFT density and solids content. Table C-3 is adapted from the relevant table provided by Dr. MacKinnon, Syncrude Canada Ltd. Since the baseline MFT in System 1 had solids content of 37.9 wt%, Table C-3 is based on 38 wt% of initial MFT solids content. After 366 d of incubation, the solids content of MFT in System 1 reached 42.5 wt% in MFT-U zone and 42.9 wt% in MFT-L zone (see Table 6-3). At these solids contents, Table C-3

shows that nearly 81 vol% of the original MFT porewater content still remains in the MFT layer.

In the CT of System 1, the baseline solids content was 57.2 wt% for "Base CT 1" and 56.5 wt% for "Base CT 2" (see Table B-6), similar to 56.6 wt% of the baseline solids content in the CT of System 4. Figure C-29 shows that the solids content of CT increased over the entire incubation time, with the most rapid solids content increase occurring in the first 60 d of incubation. The solids content of CT reached 71.4 wt% in CT-U and 69.2 wt% in CT-L at 62 d of incubation, and 73.2 wt% in CT-U zone and 73.5 wt% in CT-L zone at 366 d of incubation. The solids content increase in the CT is consistent with CT tailings volume changes over time (see Figure C-1).

A field study of the CT beneath MFT deposition test conducted by Syncrude Canada Ltd. (Shaw *et al.* 2001) showed that, over about 2.5 y of deposition period, the solids content of CT increased from 55 to 72 wt%, while the solids content of MFT increased from 33 to 41 wt%. In System 1 of this study, over 1 y of incubation, the solids content of CT increased from 56.5 wt% to 73.2 wt% in CT-U zone and to 73.5 wt% in CT-L zone, while the solids content of MFT increased from 37.9 wt% to 42.5 wt% in MFT-U zone and to 42.9 wt% in MFT-L zone (see Table 6-3). This comparison of solids contents between this work and the field study of Shaw *et al.* (2001) implies that further densification of both the MFT and CT in System 1 would be slow.

Figure C-29 shows that the upper and lower zones of CT and MFT in System 1 had similar solids content within individual CT and MFT layers,

indicating uniform densification in the CT or MFT layer. The solids content analysis also shows that no substantial mixing occurred between the MFT and CT layers in System 1. Since the lower MFT zone and upper CT zone samples in System 1 were separated by a 4 cm interval which was discarded when cutting the frozen columns (see Section 4.2), it might still be possible that at the interface of MFT and CT, some degree of mixing have occurred. The field demonstration of the CT beneath MFT deposition conducted by Syncrude Canada Ltd. in 1995 (Shaw *et al.* 2001) provided the similar conclusion; that is, the distinct CT and MFT layers were preserved but there was also a thin size of transition zone between the two layers.

*6.3.4 Physical and chemical characteristics in System 2 (Cap water/MFT/CT)*

System 2 had six columns and was also a CT beneath MFT system as in System 1. The six columns in System 2 were divided into three groups, each with different chemical amendment in the baseline CT, as shown in Table 4-5. The columns C2-1 to C2-3 had the same chemical composition in the baseline MFT and CT as in System 1, and were comparable to the columns in System 1. Columns C2-4 to C2-6 had elevated $Ca^{2+}$ and $SO_4^{2-}$ amendments in baseline CT, with extra addition of acetate in baseline CT for column C2-6 (see Table 4-5).

As discussed previously, at every cap water sampling, the existing cap water on the top of the MFT layer was completely extracted. Thus, at every sampling, the cap water is newly released water since the previous sampling.

Therefore, the chemical analysis of the cap water in System 2 can provide a time profile of released water composition at different incubation times.

Over 1 y of incubation, 9 in-situ samplings during incubation with 1 sacrificial sampling at the end of 1 y of incubation were conducted. Columns C-1, C-4, and C-6 were sacrificed. Because the tailings settling and cap water producing occurred more rapidly at early incubation times, more frequent in-situ sampling was conducted at early incubation times than at late incubation times.

6.3.4.1 Bromide tracer analysis

As in System 1, approximately 0.22 g NaBr was added to 1 L CT at column filling for System 2 (see Table 4-5). For System 2, the baseline $Br^-$ concentration in the MFT (Base MFT 3) was non-detectable (<0.5 mg/L); the baseline $Br^-$ concentration in the CT was 300 mg/L for columns C2-1 to C2-3 (Base CT 4) and 280 mg/L for columns C2-4 to C2-6 (Base CT 5 and Base CT 6) (see Table B-6). The baseline $Br^-$ concentrations in the MFT and CT of System 2 were similar to those in System 1.

Figure C-30 shows the $Br^-$ concentration in the cap water of System 2 over 352 d of incubation. The $Br^-$ concentration in the cap water of System 2 increased steadily over 352 d of incubation. As discussed in System 1, the cap water $Br^-$ concentration can indicate the proportion of MFT and CT porewaters in the cap water. Increased $Br^-$ concentration in the cap water indicates the increased portion of CT porewater. The data of $Br^-$ concentration in the cap

water of System 2 appear to match that of System 1 (see Figure C-21), which also shows slight $Br^-$ concentration increase in the cap water over time.

6.3.4.2 Calcium and magnesium concentrations

The baseline $Ca^{2+}$ concentration in the MFT of System 2 (Base MFT 3) was 24 mg/L, similar to the baseline $Ca^{2+}$ concentration in the MFT of System 1 (see Table B-6). The baseline $Ca^{2+}$ concentration in the CT for columns C2-1, C2-2, and C2-3 (Base CT 4) was 78 mg/L, similar to the baseline $Ca^{2+}$ concentration in the CT for System 1. The baseline $Ca^{2+}$ concentration in the CT for columns C2-4 and C2-5 (Base CT 5) was 99 mg/L, and for column C2-6 (Base CT 6) was 114 mg/L.

Figure C-31 shows the $Ca^{2+}$ concentration in the cap water of System 2 over 352 d of incubation. Figure C-31 shows that the $Ca^{2+}$ concentration decreased slightly in System 2 over the 352 d of incubation, indicating that the release of $Ca^{2+}$ from the CT water decreased slightly with time. The $Ca^{2+}$ concentration in the cap water of columns C2-1 to C2-3 was in the range of 17 to 32 mg/L over the 352 d of incubation, comparable to the range of 23 to 30 mg/L of $Ca^{2+}$ concentration in the cap water of System 1 over 366 d of incubation. Compared to 78 mg/L of baseline $Ca^{2+}$ concentration in the CT for columns C2-1 to C2-3 (Base CT 4), columns C2-1 to C2-3 in System 2 were effective at reducing the $Ca^{2+}$ concentration for the cap water, as was the case for System 1.

Columns C2-4 to C2-6 had $Ca^{2+}$ concentrations in the cap water ranging from 26 to 41 mg/L over 352 d of incubation, slightly higher than in columns C2-1 to C2-3. These higher concentrations of $Ca^{2+}$ in the cap water are most likely due to the higher baseline $Ca^{2+}$ concentration in the CT. Therefore, the higher $Ca^{2+}$ concentration in the baseline CT will lead to a higher $Ca^{2+}$ concentration in the cap water. Compared to 99 mg/L of baseline $Ca^{2+}$ concentration in CT for columns C2-4 and C2-5 (Base CT 5) and to 114 mg/L of baseline $Ca^{2+}$ concentration in CT for column C2-6 (Base CT 6), columns C2-4 to C2-6 in System 2 were effective at reducing the $Ca^{2+}$ concentration for the cap water.

Figure C-32 shows the $Mg^{2+}$ concentration in the cap water of System 2 over 352 d of incubation. The baseline CT for System 2 had slightly higher $Mg^{2+}$ concentrations (28 mg/L for Base CT 4, 30 mg/L for Base CT 5, and 34 mg/L for Base CT 6) than the baseline MFT for System 2 (16 mg/L for Base MFT 3). The $Mg^{2+}$ concentrations in the baseline CT and MFT for columns C2-1 to C2-3 were close to the $Mg^{2+}$ concentrations in the baseline CT and MFT for System 1.

The release of $Mg^{2+}$ into the cap water over time was quite constant during 352 d of incubation (see Figure C-32). The $Mg^{2+}$ concentration in the cap water was in the range of 14 to 21 mg/L for columns C2-1 to C2-3, and 19 to 27 mg/L for columns C2-4 to C2-6. These cap water $Mg^{2+}$ concentrations in System 2 are similar to the range of 12 to 19 mg/L in the cap water of System 1 during 366 d of incubation. Compared to the $Mg^{2+}$ concentrations in the baseline MFT

and CT, System 2 was effective at reducing the $Mg^{2+}$ concentration for the cap water.

6.3.4.3 Sulphate concentration

From Table B-6, it can be seen that the baseline $SO_4^{2-}$ concentration in the MFT for System 2 (Base MFT 3) was 23 mg/L; the baseline $SO_4^{2-}$ concentration in CT for columns C2-1 to C2-3 (Base CT 4) was 1070 mg/L, for columns C2-4 and C2-5 (Base CT 5) was 1350 mg/L, and for column C2-6 (Base CT 6) was 1660 mg/L. In comparison, the baseline $SO_4^{2-}$ concentration in the MFT of System 1 (Base MFT 1) was non-detectable (<0.5 mg/L); the baseline $SO_4^{2-}$ concentration in the CT of System 1 was 724 mg/L for Base CT 1 and 897 mg/L for Base CT 2.

Figure C-33 shows the $SO_4^{2-}$ concentration in the cap water of System 2 over 352 d of incubation. The $SO_4^{2-}$ concentrations in the cap water of System 2 showed similar trends as the $SO_4^{2-}$ concentrations in the cap water of System 1 (see Figure C-4). The $SO_4^{2-}$ concentration increased quickly at the beginning of the incubation to reach a $SO_4^{2-}$ concentration, after 3 to 7 d of incubation, of the range of 260 to 360 mg/L for columns C2-1 to C2-3, and of the range of 388 to 700 mg/L for columns C2-4 to C2-6. After this initial increase, the $SO_4^{2-}$ concentration decreased steadily to reach a low value at 49 d of incubation. After 49 d of incubation, the $SO_4^{2-}$ concentrations increased over time. The reasons for this $SO_4^{2-}$ concentration increase in the cap water were unknown.

Table B-10 shows the $S^{2-}$ analysis results on the cap water of System 2. All of the $S^{2-}$ concentration in the cap water of System 2 was non-detectable (<0.05 mg/L). In fact, due to the high concentration of $O_2$ in the headspace, any $S^{2-}$ produced from $SO_4^{2-}$ reduction by SRB would be oxidized and hence disappear from the cap water.

The columns in System 2 had different $SO_4^{2-}$ concentrations in the baseline CT (see Table 4-5). As shown in Figure C-33, the higher $SO_4^{2-}$ concentrations in the CT would likely lead to a higher $SO_4^{2-}$ concentration in the cap water. The addition of acetate in the baseline CT of column C2-6 seemed to have no obvious effect in increasing the removal of $SO_4^{2-}$ by microbial activity over the research period.

*6.3.5 Comparisons among Systems 1, 3, and 4*

System 1 (Cap water/MFT/CT) was the main research system, while Systems 3 (Cap water/MFT) and 4 (Cap water/CT) served as controls. Because the cap water properties and tailings densification are the foremost important parameters in assessing System 1 of this study, further comparisons of the major cap water parameters ($Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ and EC) and tailings solids content among the main system (System 1) and control systems (Systems 3 and 4) will be presented here.

6.3.5.1 Comparisons of $Ca^{2+}$, $Mg^{2+}$, and $SO_4^{2-}$ concentrations in the cap water

Figure C-34 shows the comparison of the $Ca^{2+}$ concentrations in the cap water of Systems 1, 3, and 4 over 1 y of incubation. The $Ca^{2+}$ concentration in the cap water of System 1 was in the range of 23 to 30 mg/L over 1 y of incubation. The $Ca^{2+}$ concentration in the cap water of System 1 was only slightly higher than in System 3, and lower than in System 4.

Figure C-35 shows the comparison of the $Mg^{2+}$ concentrations in the cap water of Systems 1, 3, and 4 over 1 y of incubation. The $Mg^{2+}$ concentrations in the cap water of System 1 were in the range of 16 to 19 mg/L over 1 y of incubation. As with $Ca^{2+}$, the $Mg^{2+}$ concentration in the cap water of System 1 was only slightly higher than in System 3, and lower than in System 4.

Figure C-36 shows the comparison of the $SO_4^{2-}$ concentrations in the cap water of Systems 1, 3, and 4 over 1 y of incubation. The $SO_4^{2-}$ concentration in the cap water of System 1 was in the range of 0 to 277 mg/L over 1 y of incubation. As with $Ca^{2+}$ and $Mg^{2+}$, the $SO_4^{2-}$ concentrations in the cap water of System 1 were higher than those of System 3 but lower than those in System 4.

It is clear from the comparisons among Figures C-34 to C-36 that, over 1 y of incubation, System 1 was effective at reducing the $Ca^{2+}$, $Mg^{2+}$, and $SO_4^{2-}$ concentrations for the cap water and could reach concentrations close to those in the MFT only system.

6.3.5.2 Comparisons of pH and electrical conductivity in the cap water

Figure C-37 shows the comparison of the pH in the cap water of Systems 1, 3, and 4 over 1 y of incubation. The pH in the cap water of all three systems was quite constant over time and was mostly in the range of 7.6 to 8.2.

Figure C-38 shows the comparison of the EC in the cap water of Systems 1, 3, and 4 over 1 y of incubation. The EC in the cap water of all three systems increased slightly with time. Over 1 y of incubation, the EC in the cap water of System 1 was in the range of 4560 to 5270 μS/cm, higher than the range of 4340 to 4670 μS/cm in the cap water of System 3, but lower than the range of 6100 to 6440 μS/cm in the cap water of System 4.

As with $Ca^{2+}$, $Mg^{2+}$, and $SO_4^{2-}$, System 1 was effective at reducing the EC for the cap water, and could reach a level close to that of System 3.

6.3.5.3 Comparison of solids content in MFT and CT

Figure C-39 shows the comparison of the solids content in the MFT between Systems 1 and 3 over 1 y of incubation. The solids content in the MFT of both Systems 1 and 3 increased steadily over the incubation time. The MFT in System 3 had consistently higher solids content than the MFT of System 1, indicating that the MFT in System 1 did not consolidate as fast as the MFT in System 3. The upper and lower zones of MFT showed similar solids content in both Systems 1 and 3 over the 1 y of incubation, indicating the uniform densification over the entire MFT layer in both systems.

Figure C-40 shows the comparison of the solids content in CT between Systems 1 and 4 over 1 y of incubation. The solids content in the CT of both Systems 1 and 4 increased steadily over incubation time, with most rapid increase occurring in the first 60 d of incubation. The CT in both Systems 1 and 4 showed similar solids content increase over 1 y of incubation, indicating the CT in System 1 consolidated as fast as the CT in System 4. The upper and lower zones of CT showed similar solids content in both Systems 1 and 4 over the 1 y of incubation, indicating the uniform densification over the entire CT layer in either system.

## 6.4 MPN analysis on MFT and CT in System 1

The MPN analyses of methanogens and SRB were conducted on MFT and CT samples of System 1 usually at an interval of every 60 d of incubation. Table B-13 lists the results of MPN analysis. Table B-13 shows that both the MFT and CT in System 1 were rich in methanogens and SRB. The activity of these groups of microorganisms will contribute to the production of $CH_4$ by methanogens and the reduction of $SO_4^{2-}$ by SRB.

### *6.4.1 MPN of methanogens*

Figure C-41 shows the log (MPN) of methanogens over 1 y of incubation in System 1. For the methanogens in System 1, the MPN was for the most part in the range of $10^3$ to $10^5$/g (of dry solids) in MFT and of $10^2$ to $10^4$/g (of dry

solids) in CT. The upper and lower zones of MFT showed similar MPN values of methanogens in System 1 over 1 y of incubation. The MFT had higher MPN of methanogens than the CT over 1 y of incubation. Fedorak *et al.* (2000) reported that the MPN for Syncrude MFT was mostly in the range of $10^5$ to $10^7$/g (of dry solids) over 1 y of incubation. The MFT in System 1 was placed above a layer of CT, thus different from the MFT in Fedorak *et al.* (2000). The low MPN value of the MFT in System 1 as compared to that in Fedorak *et al.* (2000) may be due to the high $SO_4^{2-}$ concentrations (> 20 mg/L) in the MFT porewater of System 1 (see Figure C-4). The MPN in the CT of System 1 was lower than the MPN of MFT. The high $SO_4^{2-}$ concentration in the CT may account for the low MPN of methanogens (see Figure C-4). As discussed previously, the presence of high concentration of $SO_4^{2-}$ will stimulate the activity of SRB and hence inhibit the methanogen activity.

### *6.4.2 MPN of SRB*

Figure C-42 shows the log (MPN) of SRB over 1 y of incubation in System 1. For the SRB in System 1, the MPN was for the most part in the range of $10^5$ to $10^6$/g (of dry solids) in MFT and of $10^4$ to $10^5$/g (of dry solids) in CT. The upper and lower zones of MFT showed similar MPN of SRB in System 1 over 1 y of incubation. As shown in Figure C-4, the CT porewater had consistently higher $SO_4^{2-}$ concentration than the MFT porewater in System 1. From the high $SO_4^{2-}$ concentration, the CT in System 4 should favour the

activity of SRB and have high MPN of SRB. The MFT, however, had consistently higher MPN of SRB than the CT in System 1 over 1 y of incubation. The reasons for the higher MPN of SRB in the MFT than in the CT cannot be identified. The larger MPN values of SRB in the MFT should lead to more $SO_4^{2-}$ reduction in the MFT than in the CT of System 1. This appears to be supported by the AVS analysis, which shows larger AVS values in the MFT than in the CT at 121 d of incubation in System 1 (see Table 6-16). Holowenko (2000) reported that the MPN of SRB was for the most part in the range of $10^5$ to $10^7$/g (of dry solids) for Syncrude MFT.

## 7.0 Summary

### 7.1 Total volume change (Vt) in Systems 3, 4, and 1

For System 3, over 1 y of incubation, the total volume of the MFT only deposit expanded initially and reached a maximum Vt(3) value in the range of 12.3 to 13.5 vol% among the columns at 90 d of incubation, and then the total volume decreased and reached a Vt(3) value of 6.9 vol% at 365 d of incubation.

For System 4, over 1 y of incubation, the total volume change of the CT only deposit was small, with the Vt(4) values in the range of 0.0 to –0.7 vol% among the columns.

For System 1, over 1 y of incubation, the total volume change (Vt(1)) of the CT beneath MFT deposit was between –8.8 vol% and 2.8 vol% among the columns.

### 7.2 Total tailings volume change (Vtt) in Systems 3, 4, 1, and 2

For System 3, over 1 y of incubation, the total tailings volume of the MFT only deposit increased initially and reached a maximum Vtt(3) value between 3.6 and 4.6 vol% among the columns at 77 d of incubation. After this time, the total tailings volume decreased steadily and reached a Vtt(3) value of 0 vol% at about 110 d of incubation, and of –8.8 vol% at 365 d of incubation.

For System 4, over 1 y of incubation, the total tailings volume of the CT only deposit decreased steadily. The most rapid decrease in total tailings volume occurred in the first 60 d of incubation, with a Vtt(4) value between -27.7 and

-28.6 vol% among the columns at 61 d of incubation and of -31.9 vol% at 365 d of incubation.

For System 1, over 1 y of incubation, the total tailings volume of the CT beneath MFT deposit decreased steadily. The most rapid decrease in total tailings volume occurred in the first 60 d of incubation, with a Vtt(1) value between –17 and –21.2 vol% among the columns at 62 d of incubation and between –22.4 and –26.2 vol% among the columns at 366 d of incubation.

For System 2, the total tailings volume of the CT beneath MFT deposit decreased steadily. The most rapid decrease in total tailings volume occurred in the first 60 d of incubation, with a Vtt(2) value between –18.1 to –21.5 vol% among the columns at 49 d of incubation and between –22.5 to –26.8 vol% among the columns at 352 d of incubation. The total tailings volume change showed no certain effects of the different chemical dosage in CT on the total tailings densification rate over the experiment period.

### 7.3 Individual MFT and CT tailings volume change ($V_{MFT}$ and $V_{CT}$) in Systems 1 and 2

The MFT layer in System 1 did not show a definite tailings volume decrease but rather a fluctuating value over 1 y of incubation. The trapped gas bubbles in the MFT and errors caused in determining the Geo-grid position may have contributed to this fluctuating tailings volume data of the MFT.

The CT layer in System 1 consolidated to a larger extent than the MFT layer over 1 y of incubation. Most of the tailings volume decrease of the CT

occurred in the first 60 d of incubation, with a $V_{CT(1)}$ value of -29.7 vol% at 62 d of incubation and of -33.6 vol% at 366 d of incubation.

The MFT layer in System 2 expanded slightly in the first 2 wk of incubation, with a maximum expansion occurring at a $V_{MFT(2)}$ value of 2.9 vol% at 14 d of incubation. Then the MFT started to densify steadily, with a $V_{MFT(2)}$ value in the range of -1.5 to 0.4 vol% at 49 d of incubation and of -6.2 to -14.9 vol% at 352 d of incubation among the columns in System 2. The tailings volume change of the MFT showed no certain effects of the different chemical dosages in baseline CT on the MFT densification rate over the experiment period.

As in System 1, the CT in System 2 consolidated to a larger extent than the MFT. The tailings volume of CT decreased steadily over 1 y of incubation. Most of the CT tailings volume decrease occurred in the first 60 d of incubation, with a $V_{CT(2)}$ value in the range of –28.3 to -31.7 vol% at 49 d of incubation and in the range of –31.8 to -37.4 vol% at 352 d of incubation among the columns in System 2. The tailings volume change of the CT showed no certain effects of the different chemical dosages in baseline CT on the CT densification rate over experiment period.

### 7.4 Comparisons of total and individual tailings volume changes (Vtt, $V_{MFT}$, and $V_{CT}$) among Systems 1, 2, 3 and 4

For the total tailings volume change, over 1 y of incubation, System 4 had the highest total tailings volume reduction with a Vtt(4) value of –31.9 vol%

at 365 d of incubation; then followed by System 1 with a Vtt(1) value of –22.4 to –26.2 vol% among the columns at 366 d of incubation; and System 3 had the lowest total tailings volume reduction with a Vtt(3) of –8.8 vol% at 365 d of incubation. The decrease in total tailings volume of Systems 1 and 4 occurred most rapidly in the first 60 d of incubation and reached a quite stable level after 1 y of incubation, while the total tailings volume in System 3 showed a further trend of decrease after 1 y of incubation.

For the individual MFT tailings volume change, Systems 2 and 3 showed similar MFT tailings volume reduction over incubation, with a $V_{MFT(2)}$ value in the range of –6.2 to –14.9 vol% among the columns at 352 d of incubation in System 2 and a $V_{MFT(3)}$ value of –8.8 vol% at 365 d of incubation in System 3.

For the individual CT tailings volume change, Systems 2 and 4 showed similar CT tailings volume reduction over incubation, with a $V_{CT(2)}$ value in the range of –31.8 to -37.4 vol% among the columns at 352 d of incubation in System 2 and a $V_{CT(4)}$ value of –31.9 vol% at 365 d of incubation in System 4.

### 7.5 Cap water volume (Vcw) in Systems 3, 4, and 1

In System 3, the cap water was produced steadily over 1 y of incubation. In System 4, most of the cap water was produced in the first 60 d of incubation. In System 1, most of the cap water was produced in the first 60 d of incubation, with a Vcw(1) value in the range of 17.3 to 20.2% at 62 d of incubation and of

17.4 to 24.7 vol% at 365 d of incubation among the columns. Systems 1 and 4 produced more cap water than System 3 over 1 y of incubation.

### 7.6 Cap water producing rate (Rcw) in Systems 3, 4, and 1

For System 3, the cap water producing rate among the columns reached a maximum value of approximately 17 mL/d in 30 d of incubation, and then decreased slowly to a value of 0.8 mL/d at 365 d of incubation.

For System 4, the cap water producing rate among the columns decreased from an initial value of approximately 200 mL/d to a value between 7.5 and 9.4 mL/d at 61 d of incubation, and to approximately 0 mL/d by 212 d of incubation.

For System 1, the cap water producing rate among the columns decreased from an initial value of approximately 200 mL/d to a value between 6.4 and 9.4 mL/d at 62 d of incubation, and to approximately 0 mL/d by 244 d of incubation, indicating that further dewatering of System 1 would be slow.

### 7.7 Tedlar bag gas volume in Systems 1, 3, and 4

In Systems 1 and 4, the Tedlar bag gas volumes were small. In System 1, only 10 to 60 mL of Tedlar bag gas was collected in the sacrificed columns over 366 d of incubation. In System 4, 17 to 308 mL of Tedlar bag gas was collected in the sacrificed columns over 365 d of incubation. No obvious gas evolution was visually observed in the columns of Systems 1 or 4.

In System 3, the Tedlar bag gas volume was higher and showed a large variation among the different columns. The Tedlar bag gas volume in the sacrificed columns in System 3 was in the range of 20 to 2030 mL over 365 d of incubation. Obvious gas evolution in the columns of System 3 was visually observed.

**7.8 GC analysis on the Tedlar bag gas in Systems 1, 3, and 4**

System 1 had for the most part a higher production of $CH_4$ than System 4, but a lower production of $CH_4$ than System 3. In System 1, the $CH_4$ concentration in the Tedlar bag gas increased steadily from 0.04 vol% at 29 d of incubation to 4.57 vol% at 213 d of incubation. In System 3, the $CH_4$ concentration in the Tedlar bag gas increased steadily from 0.39 vol% at 28 d of incubation to 20.70 vol% at 212 d of incubation. In System 4, the $CH_4$ concentrations in the Tedlar bag gas were quite low, only between 0 to 0.29 vol% over 1 y of incubation.

The $CO_2$ concentrations in the Tedlar bag gas of Systems 1, 3, and 4 increased steadily and reached a maximum level of approximately 4 to 8 vol% over 1 y of incubation. The measured $CO_2$ concentrations by GC in the Tedlar bag gas of System 1 were comparable with the calculated $CO_2$ concentrations in the column headspace gas of System 1 by Henry's law.

In the column headspaces of Systems 1, 3, and 4, low concentrations (for the most part, <1.0 vol%) of $H_2S$ and $C_2H_4$ were occasionally detected.

### 7.9 In-situ headspace gas GC analysis in System 2

Compared to the Tedlar bag gas analysis in System 1, the in-situ headspace gas analysis in System 2 showed low $CH_4$ and $CO_2$ concentrations and a high $O_2$ concentration. Low concentrations (<1.0 vol%) of $H_2S$ and $C_2H_4$ were detected occasionally.

The in-situ headspace analysis showed that the different chemical amendments in CT had little effects on the production and composition of headspace gases over 1 y of incubation.

Because of the disturbance and air contamination introduced in the in-situ headspace gas analysis, the in-situ gas analysis data of System 2 should be used with caution. The Tedlar bag gas analysis in Systems 1, 3, and 4 will be more reliable and valid in characterising the gas evolution from MFT or CT deposits.

### 7.10 Bromide tracer analysis and the dilution effect in System 1

In the MFT, over 1 y of incubation, the $Br^-$ concentration increased steadily from initially non-detectable levels (<0.5 mg/L) to 130 mg/L in MFT-U zone and 210 mg/L in MFT-L zone at 62 d of incubation, and remained quite stable in the range of 120 to 150 mg/L for MFT-U zone and 160 to 210 mg/L for MFT-L zone for the remainder of the incubation.

In the CT, over 1 y of incubation, the $Br^-$ concentration was quite constant and for the most part in the range of 220 to 280 mg/L, close to the baseline $Br^-$ concentration in the CT.

In the cap water, the $Br^-$ concentration increased quickly to 82 mg/L in the first 29 d of incubation, and remained in the range of 70 to 110 mg/L over the remaining incubation.

The mass balance calculations of $Br^-$ showed that over 1 y of incubation, the $Br^-$ recovery (relative to the $Br^-$ mass at time 0) in System 1 was for the most part in the range of 92 to 108%. This indicates that the $Br^-$ is a stable tracer with almost 100% recovery in the MFT and CT materials over incubation time.

The $Br^-$ in the MFT and cap water originated from the underlying CT release water. The presence of $Br^-$ in the MFT and cap water indicates the presence of CT water. Based on $Br^-$ concentration analysis, it was shown that both the cap water and MFT porewater contained large percentages of CT water during the incubation, with a CT water portion of 41 vol% in cap water, 56 vol% in MFT-U zone, and 67 vol% in MFT-L zone at 366 d of incubation.

The estimation of dilution effect ($P_d$%) showed that dilution effect played a large role at reducing the concentrations of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and the EC for the cap water in System 1.

### 7.11 Calcium and magnesium concentrations in System 1

In the MFT, the $Ca^{2+}$ concentration decreased slightly, with the $Ca^{2+}$ concentration of 17 mg/L in the MFT-U zone and 19 mg/L in the MFT-L zone

at 15 d of incubation, and 13 mg/L in the MFT-U zone and 14 mg/L in the MFT-L zone at 366 d of incubation. The MFT-U zone had slightly lower $Ca^{2+}$ concentrations than the MFT-L zone.

In the CT, the largest drop in $Ca^{2+}$ concentration occurred during the first 30 d of incubation, with a $Ca^{2+}$ concentration of 29 mg/L in CT-U zone and 23 mg/L in CT-L zone at 29 d of incubation, and 21 mg/L in CT-U zone and 18 mg/L in CT-L zone at 366 d of incubation. The two CT-U and CT-L zones had similar $Ca^{2+}$ concentration over 1 y of incubation. The CT had higher $Ca^{2+}$ concentrations than the MFT.

In the cap water layer, the $Ca^{2+}$ concentration was quite constant and in the range of 23 to 30 mg/L over 1 y of incubation. The cap water had $Ca^{2+}$ concentration higher than in the MFT and close to that in the CT.

The mass balance calculations of $Ca^{2+}$ showed that over 1 y of incubation, more than 50% of the original $Ca^{2+}$ mass was removed from the water by some mechanisms. The ratios of $[Ca^{2+}]\times[CO_3^{2-}]$ to Ksp in all zones of System 1 over 1 y of incubation are in the range of 3 to 12, indicating that the precipitation of $CaCO_3$ is a possible route for the removal of $Ca^{2+}$ in System 1.

The $Mg^{2+}$ concentration in the MFT was quite constant (ranging from 11 to 16 mg/L) over 1 y of incubation. The $Mg^{2+}$ concentration in the CT decreased most rapidly in the early incubation and reached 14 mg/L in CT-U zone and 15 mg/L in CT-L zone at 29 d of incubation. After this initial decrease, the $Mg^{2+}$ concentration in the CT was quite stable. The $Mg^{2+}$ concentration in the cap

water was in the range of 16 to 19 mg/L over 1 y of incubation and higher than in either MFT or CT.

### 7.12 Sulphate concentration in System 1

In the MFT, the $SO_4^{2-}$ concentration increased rapidly from essentially 0 mg/L of baseline concentration to 115 mg/L in MFT-U and 226 mg/L in MFT-L at 3 d of incubation, then decreased mostly in the first 30 d of incubation with 31 mg/L in MFT-U zone and 87 mg/L in MFT-L zone at 29 d of incubation. After that time, $SO_4^{2-}$ concentration remained relatively constant and reached 44 mg/L in MFT-U zone and 60 mg/L in MFT-L zone at 366 d of incubation. The MFT-U zone had for the most part lower $SO_4^{2-}$ concentrations than the MFT-L zone.

In the CT, the largest decrease in $SO_4^{2-}$ concentration occurred during the first 30 d of incubation, with $SO_4^{2-}$ concentrations of 336 mg/L in CT-U zone and 260 mg/L in CT-L zone at 29 d of incubation. After that time, although showing some fluctuation, the $SO_4^{2-}$ concentration for the most part decreased, and reached 21 mg/L in CT-U zone and 23 mg/L in CT-L zone at 366 d of incubation, corresponding to more than 95% $SO_4^{2-}$ concentration reduction. The upper and lower zones of CT showed similar $SO_4^{2-}$ concentration.

The MFT had lower $SO_4^{2-}$ concentration than the CT over 1 y of incubation. However, after 1 y of incubation, both the MFT and CT approached similar $SO_4^{2-}$ concentration in the range of 21 to 60 mg/L.

In the cap water, the $SO_4^{2-}$ concentration increased initially and reached a maximum value of 235 mg/L at 15 d of incubation. The $SO_4^{2-}$ concentration

dropped to 73 mg/L at 62 d of incubation, and then remained quite stable until 213 d of incubation at 52.6 mg/L. After 244 d of incubation, the $SO_4^{2-}$ concentration in the cap water increased unexpectedly.

The mass balance calculations of $SO_4^{2-}$ showed that, after 1 y of incubation, more than 80% of the original $SO_4^{2-}$ mass was removed from the water by some mechanisms in System 1.

**7.13 pH and electrical conductivity in System 1**

The pH in the cap water and porewaters of MFT and CT was for the most part between 7.6 and 8.3 over 1 y of incubation. The cap water had consistently lower pH values than the MFT and CT porewaters.

In the MFT, the EC increased slightly over time and reached 4870 μS/cm in MFT-U zone and 4940 μS/cm in MFT-L zone at 366 d of incubation. In the CT, the EC showed a rapid decrease in the first 30 d of incubation. Following this initial rapid decrease, the EC in the CT increased slightly over time and reached 5350 μS/cm in CT-U zone and 5190 μS/cm in CT-L zone at 366 d of incubation. The two CT-U and CT-L zones showed similar EC during the incubation. In the cap water, the EC increased with time and was in the range of 4560 to 5330 μS/cm during 1 y of incubation.

**7.14 Solids content in System 1**

In the MFT, the solids content increased steadily over 1 y of incubation, and reached 42.5 wt% in MFT-U zone and 42.9 wt% in MFT-L zone at 366 d of

incubation. The MFT-U and MFT-L in the MFT showed similar solids content over time, indicating uniform densification over the entire MFT layer. The MFT solids content data are believed to be more reliable than the MFT tailings volume change ($V_{MFT(1)}$) in characterising the densification behaviour of MFT in System 1.

At 366 d of incubation, the solids contents of MFT in System 1 reached 42.5 wt% in upper zone of MFT and 42.9 wt% in the lower zone of MFT. At these solids contents, nearly 81 vol% of porewater (relative to the original porewater content) still remains in the MFT.

In the CT, the solids content increased most rapidly in the first 60 d of incubation, reaching 71.4 wt% in CT-U zone and 69.2 wt% in CT-L zone at 62 d of incubation. After that time, the CT solids content increased continuously over the remaining incubation and reached 73.2 wt% in CT-U and 73.5 wt% in CT-L at 366 d of incubation. The upper and lower zones of CT showed similar solids content over time, indicating uniform densification over the entire CT layer. The solids content analysis also showed that no obvious mixing occurred between the MFT and CT layers in System 1.

### 7.15 Bromide tracer analysis in System 2

The $Br^-$ concentration in the cap water of System 2 increased over 352 d of incubation, indicating the increased portion of CT porewater in the cap water

over incubation time. At 352 d of incubation, the $Br^-$ concentration in the cap water of System 2 reached 160 to 200 mg/L in the three sacrificed columns.

### 7.16 Calcium and magnesium concentrations in the cap water of System 2

In the cap water of System 2, the $Ca^{2+}$ concentration decreased slightly in System 2 over 352 d of incubation, indicating that the release of $Ca^{2+}$ from the CT beneath MFT deposit was slightly decreasing over time. The $Ca^{2+}$ concentration in the cap water of columns C2-1 and C2-3 was in the range of 17 to 32 mg/L during 352 d of incubation, comparable to the range of 23 to 30 mg/L of $Ca^{2+}$ concentration in the cap water of System 1 over 366 d of incubation. Compared to the baseline $Ca^{2+}$ concentration in the MFT and CT, as in System 1, columns C2-1 to C2-3 in System 2 were effective at reducing $Ca^{2+}$ concentration for the cap water. The higher concentration of $Ca^{2+}$ in the CT at column filling lead to higher $Ca^{2+}$ concentration in the cap water, as shown in columns C2-4 to C2-6.

The $Mg^{2+}$ concentration in the cap water of System 2 was quite constant during 352 d of incubation and in the range of 14 to 21 mg/L for columns C2-1 to C2-3, similar to the range of 12 to 19 mg/L in the cap water of System 1 during 366 d of incubation. Compared to the baseline $Mg^{2+}$ concentration in the MFT and CT, System 2 was effective at reducing $Mg^{2+}$ concentration for the cap water.

### 7.17 Sulphate concentration in the cap water of System 2

The $SO_4^{2-}$ concentration during incubation in the cap water of System 2 showed a similar pattern as the $SO_4^{2-}$ concentration in the cap water of System 1. That is, in the cap water of System 2, the $SO_4^{2-}$ concentration increased initially and reached a high $SO_4^{2-}$ concentration in 3 to 7 d of incubation, then decreased and reached the lowest value at 49 d of incubation. After that time, the $SO_4^{2-}$ concentration increased again with time. The reasons for this $SO_4^{2-}$ concentration increase were not clear.

The higher concentration of $SO_4^{2-}$ in the baseline CT caused higher $SO_4^{2-}$ concentration in the cap water. However, the addition of acetate seemed to have no obvious effects on the removal rate of $SO_4^{2-}$ over the experiment period.

### 7.18 Comparisons of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ concentrations in the cap water of Systems 1, 3, and 4

The concentrations of $Ca^{2+}$, $Mg^{2+}$, and $SO_4^{2-}$ in the cap water of System 1 were slightly higher than in System 3, but much lower than in System 4, indicating that System 1 was effective at reducing $Ca^{2+}$, $Mg^{2+}$, and $SO_4^{2-}$ concentrations for the cap water.

### 7.19 Comparisons of pH and electrical conductivity in the cap water of Systems 1, 3, and 4

The pH values in the cap water of Systems 1, 3, and 4 were similar, and mostly in the range of 7.6 to 8.2 during 1 y of incubation. The EC in the cap

water of Systems 1, 3, and 4 increased slightly over time. During 1 y of incubation, the EC in the cap water of System 1 was in the range of 4560 to 5270 μS/cm, higher than in System 3 with the range of 4340 to 4670 μS/cm, but lower than in System 4 with the range of 6100 to 6440 μS/cm. System 1 was effective at reducing EC for the cap water.

### 7.20 Comparisons of solids content of MFT and CT among Systems 1, 3, and 4

The solids content in the MFT of both Systems 1 and 3 increased steadily over the incubation time. The MFT in System 3 had consistently higher solids content than in the MFT of System 1, indicating that the MFT in System 1 did not consolidate as fast as the MFT in System 3. The upper and lower zones of MFT showed similar solids content in either System 1 or 3 over 1 y of incubation, indicating the uniform densification rate over the entire MFT layer in either system.

The solids content in the CT of both Systems 1 and 4 increased steadily over incubation time, with largest rapid increase occurred in the first 60 d of incubation. The CT in both Systems 1 and 4 showed similar solids content increase over the 1 y of incubation, indicating that the CT in System 1 consolidated at a rate comparable to the CT in System 4. The upper and lower zones of CT showed similar solids content in either System 1 or 4 over the 1 y of incubation, indicating the uniform densification rate over the entire CT layer in either system.

### 7.21 MPN analysis on the MFT and CT in System 1

The MFT and CT in System 1 were both rich in methanogens and SRB. The MPN for methanogens was for the most part in the range of $10^3$ to $10^5$/g (of dry solids) in MFT and $10^2$ to $10^4$/g (of dry solids) in CT over 1 y of incubation. The MPN for SRB was for the most part in the range of $10^5$ to $10^6$/g (of dry solids) in MFT and $10^4$ to $10^5$/g (of dry solids) in CT over 1 y of incubation.

The upper and lower zones of MFT showed similar MPN values of SRB and methanogens.

## 8.0 Conclusion

In order to optimise tailings management, the oil sands industry has implemented CT process to handle the fluid portion (fine tailings) of its extraction tailings. The co-disposal approach using CT placed beneath MFT was proposed to improve the CT release water quality and the solids densification rates of both CT and MFT. The CT beneath MFT deposit (System 1) was assessed by comparing to the control systems (Systems 3 and 4). In the CT beneath MFT deposit, over 1 y of incubation, the cap water had improved water quality and both the MFT and CT showed steady densifications that were comparable to the control systems, as specified below:

(i) Over 1 y of incubation, the concentrations of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and the electrical conductivity in the cap water of System 1 were only slightly higher than in System 3 but much lower than in System 4, with values of 27 mg/L, 18 mg/L, 271 mg/L, and 5150 µS/cm at the end of 1 y of incubation, respectively. The mass balance calculations of $Ca^{2+}$ and $SO_4^{2-}$ showed that, after 1 y of incubation, over 50% of the original $Ca^{2+}$ mass and 80% of the original $SO_4^{2-}$ mass were removed from the water of System 1. System 1 was therefore effective at reducing the concentrations of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and the EC for the cap water.

(ii) Over 1 y of incubation, the solids content of the MFT in the CT beneath MFT deposit increased steadily from the original 37.9 wt% to 42.5 wt% in the upper zone of MFT and to 42.9 wt% in the lower zone of MFT. This solids content increase was slightly smaller than the solids content increase in

the MFT of control system. The upper and lower zones of the MFT in System 1 showed similar solids content increase over incubation time.

(iii) Over 1 y of incubation, the solids content of the CT in the CT beneath MFT deposit increased from the original 56.5 wt% to 73.2 wt% in the upper zone of CT and to 73.5 wt% in the lower zone of CT. The overall solids content increase of CT in System 1 was slightly larger than the solids content increase in the control system. The upper and lower zones of the CT in System 1 showed similar solids content increase over incubation time.

(iv) Over 1 y of incubation, the MFT and CT in the CT beneath MFT deposit were rich in methanogens and SRB. The activity of the SRB and methanogens in the MFT and CT resulted in $SO_4^{2-}$ reduction and $CH_4$ production.

(v) In decreasing the concentrations of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and the electrical conductivity for the cap water of the CT beneath MFT deposit, the simple mixing between the CT water and MFT water played a large role due to the dilution effect between the two waters.

The results indicate that the CT beneath MFT deposition is a beneficial disposal scheme for tailings management.

# 9.0 Recommendations

## 9.1 In-situ headspace gas analysis in System 2

In System 2, the in-situ headspace gas analysis was accompanied by regular cap water sampling. The void created by the removed cap water was filled with $N_2$. This procedure will cause severe air contamination in the column headspace. Therefore, the in-situ headspace gas analysis in System 2 should be used with caution.

To avoid the severe air contamination and increase the reliability of the in-situ headspace gas analysis, it is recommended to set up some extra columns to particularly allow for in-situ headspace gas analysis without accompanying the regular cap water extraction.

## 9.2 AVS analysis

The results of AVS analysis can tell, with some degree of confidence, the sinks of $SO_4^{2-}$ reduction by SRB. The AVS analysis only used approximately 3 g of MFT or CT samples. Due to the heterogeneous nature and large spatial variation of MFT and CT materials, the AVS results should therefore be used with caution. Further, more AVS data at different incubation times may be desired.

## 9.3 Trapped gas analysis

The trapped gas analysis in this project was designed for qualitative purpose of detecting mainly the presence or absence of $CH_4$. However, if a

quantitative analysis is required on the trapped gas in MFT and CT, the MFT and CT samples should be sent to the commercial laboratory with the capacity of quantitative analysis of trapped gas. Also a semi-quantitative analysis of trapped gas may be achieved by calibrating the results of this self-developed method with the results of commercial quantitative method.

### 9.4 The MFT to CT ratio for the CT beneath MFT deposition

System 1 used MFT to CT ratio of 1:2 (by volume). As discussed above, the dilution effect ($P_d$ %, Table 6-12) played a large role at reducing the concentrations of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and the EC for the cap water in System 1. It is thus expected that the increase in MFT to CT ratio will produce cap water with further lowered concentrations of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and EC values. On the other hand, since the concentrations of $Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$, and the EC in the cap water of System 1 have already reached a low level close to that in System 3 (the MFT only system), further substantial increase in MFT to CT ratio will not improve the cap water quality to a further large extent. Therefore, the MFT to CT ratios of 1:2 to 1:1 (by volume) may be a suitable range for the CT beneath MFT deposition.

## 10.0 References


ACR (Alberta Chamber of Resources), 1995. Canada's oil sands industry: yesterday, today and tomorrow. Prepared by National Task Forces on Oil Sands Strategies of the Alberta Chamber of Resources. Alberta.

AERCB (Alberta Energy Resources Conservation Board), 1984. Oil Sands Bitumen Extraction Process Evaluation, Volume 1, Summaries Phases I and II. Calgary. Alberta.

APHA (American Public Health Association), American Water Works Association, and Water Environment Federation, 1995. Standard Methods for the Examination of Water and Wastewater. 19th edition. Washington. D.C.

AWWA (American Water Works Association), 1999. Water Quality and Treatment, McGraw-Hill, Inc. 5th Edition. New York.

Duan W.M., Coleman M.L., and Pye K., 1997. Determination of Reduced Sulfur Species in Sediments---an Evaluation and Modified Technique. Chemical Geology, **141:**185-194.

Fedorak P.M., Coy D.L., Dudas M.J., Renneberg A.J., and Salloum M.J., 2000. Role of Microbiological Processes on Sulfate-enriched Tailings Deposits. Department of Biological Sciences. University of Alberta. Edmonton. Alberta.

Fedorak P.M., Coy D.L., Salloum M.J., and Dudas M.J., 2002. Methanogenic Potential of Tailings Samples from Oil Sands Extraction Plants. Can. J. Microbiol. **48**: 21-33.

FTFC (Fine Tailings Fundamentals Consortium), 1996. Meeting the Challenge-Solutions for Managing Oil Sands Tailings 1989-1995. Edmonton. Alberta.

FTFC (Fine Tailings Fundamentals Consortium), 1995. Advances in Oil Sands Tailings Research. Alberta Department of Energy. Oil Sands and Research Division. Edmonton. Alberta.

Gerard A. B., Loch J.P.G., Heijdt L.M., and Zwolsman J.J.G., 1998. Vertical Distribution of Acid-Volatile Sulfide and Simultaneously Extracted Metals in a Recent Sedimentation Area of the River Meuse in the Netherlands. Environmental Toxicology and Chemistry, **17:** 758-763.

Gray N.F., 1989. Biology of Wastewater Treatment. Oxford University Press. Oxford. England.

Holowenko F.M., 2000. Methanogenesis and Fine Tailings Waste from Oil Sand Extraction: a Microcosm-based Laboratory Examination. M.S. Thesis. University of Alberta. Edmonton. Alberta.

Hopp V. and Hennig I., 1983. Handbook of Applied Chemistry, Hemisphere Publishing Corporation. New York.

Kasperski K.L., 1992. A Review of Properties and Treatment of Oil Sands Tailings. AOSTRA Journal of Research. **8:** 11-53.

Kennedy G. L., Everett J.W., Dewers T., Pickins W., and Edwards D., 1999. Application of Mineral Iron and Sulfide Analysis to Evaluate Natural Attenuation at Fuel Contaminated Site. Journal of Environmental Engineering. **125**: 47-56.

Lasorsa B. and Casas A., 1996. A Comparison of Sample Handling and Analytical Methods for Determination of Acid Volatile Sulphides in Sediment. Marine Chemistry. **52:** 211-220.

Levett P.N., 1991. Anaerobic Microbiology: a Practical Approach. Oxford University Press. Oxford. England.

List B.R. and Lord E.R.F., 1997. Syncrude's Tailings Management Practices from Research to Implementation. CIM Bulletin. **90** (1010): 39-44.

MacKinnon M.D., 1989. Development of the Tailings Pond at Syncrude's Oil Sands Plant: 1978-1987. AOSTRA Journal of Research, **5**: 109-133

MacKinnon M.D., Matthews J.G., Shaw W.H., and Cuddy R.G., 2000. Water Quality Issue Associated with Implementation of Composite Tailings (CT) Technology for Managing Oil Sands Tailings. Proceedings of SWEMP 2000. Calgary. Alberta.

MacKinnon M. D. and Sethi A., 1993. A Comparison of the Physical and Chemical Properties of the Tailings Ponds at the Syncrude and Suncor Oil Sands Plants. Proceedings of Fine Tailings Symposium: "Oil Sands---Our Petroleum Future". Edmonton. Alberta.

Maier R.M., Ian L.P., and Charles P.G., 2000. Environmental Microbiology. Academic press. San Diego. California.

Matthews J.G., Shaw W.H., MacKinnon M.D., and Cuddy R.G., 2000. Development of Composite Tailings Technology at Syncrude Canada. Proceedings of SWEMP 2000. Calgary. Alberta.

Mikula R.J., Kasperski K.L., Burns R.D., and MacKinnon M.D., 1996. Nature and Fate of Oil Sands Fine Tailings. Advances in Chemistry Series 251. American Chemical Society. Washington. DC.

Morse J. W., Millero F.J., Cornwell J.C., and Rickard D., 1987. The Chemistry of the Hydrogen Sulfide and Iron Sulfide Systems in Natural Waters. Earth-Science Reviews, **24:** 1-42.

Nazaroff W.W. and Alvarez-Cohen L., 2001. Environmental Engineering Science. John Wiley & Sons, Inc. New York.

Postgate J.R., 1984. The Sulphate-Reducing Bacteria. Cambridge University Press. Cambridge.

Sawyer C.N., McCarty P.L., and Parkin G.F., 1994. Chemistry for Environmental Engineering. McGraw-Hill, Inc. 4th edition. New York.

Schlesinger H.W., 1997. Biogeochemistry---an analysis of global change. 2nd edition. Academic Press. San Diego. California.

Shaw B., Cuddy G., MacKinnon M., Dawson R., and Kwan K., 2001. Composite Tailings beneath Mature Fine Tailings Deposit: Summary of Laboratory and Field Work 1995 through 2000. Syncrude Canada Ltd. Report.

Shaw B., Cuddy G., McKenna G., and MacKinnon M., 1996. Non-segregating Tailings: 1995 NST Field Demonstration. Syncrude Canada Ltd. Report.

Snoeyink V.L. and Jenkins D., 1980. Water Chemistry. John Wiley & Sons, Inc. New York.

Syncrude Canada Ltd., 2000a. Syncrude Fact Book. Fort McMurray. Alberta.

Syncrude Canada Ltd., 2000b. Securing Canada's Energy Future. Fort McMurray. Alberta.

Tang J., 1997. Fundamental Behaviour of Composite Tailings. M.Sc. Thesis. University of Alberta. Edmonton. Alberta.

VCI (Varian Chrompack International), 1999. User Manual Micro-GC Portable Model CP-2003P. Walnut Creek. California.

Xiaomei L. and Yongsheng F., 1995. Dewatering Fine Tails by Evaporation: a Mathematical Modeling Approach. Report to Syncrude Canada Ltd., Edmonton Research Centre. Edmonton. Alberta.

# Appendices

# Appendix A. Methods and materials

**Table A-1. GC external calibrations (using the GC parameters at Table 4-2).**

| Gas/GC channel/ Date | $t_R$ (min) | Calibration concentration at each level (vol%) | | | | Calibration curve (linear) | $r^2$ |
|---|---|---|---|---|---|---|---|
| | | Level 1 | Level 2 | Level 3 | Level 4 | | |
| $O_2$/A/27 June-02 | 0.53 | 30.0 | 15.0 | 5.0 | 1.0 | Y=3.67(e+004) x | 0.9998 |
| $N_2$/A/18 June-02 | 0.56 | 100.0 | 75.0 | 50.0 | 25.0 | Y=3.63(e+004) x | 0.9999 |
| $CH_4$/A/03 July-02 | 0.81 | 80.0 | 50.0 | 20.0 | 1.0 | Y=3.58(e+004) x | 0.9989 |
| $CO_2$/B/24 June-02 | 0.44 | 20.0 | 10.0 | 5.0 | 1.0 | Y=1.81(e+005) x | 0.9986 |
| $C_2H_4$/B/17 Sep.-02 | 0.55 | 10.0 | 5.0 | 2.0 | 0.1 | Y=1.95(e+005) x | 0.9991 |
| $H_2S$/B/31 Oct.-02 | 1.30 | 5.0 | 3.0 | 1.0 | | Y=1.73(e+005) x | 0.9934 |

1). $t_R$=retention time.
2). $r^2$=the determination coefficient.
3). Y=the area accounts, and x is the gas concentration (vol%).
4). For $H_2S$, only three-level calibration was used.

**Table A-2. GC verifications (using the GC parameters at Table 4-2).**

| Date | S/M | Concentration (vol%)/GC channel | | | | | |
|---|---|---|---|---|---|---|---|
| | | $N_2$/A | $O_2$/A | $CH_4$/A | $CO_2$/B | $H_2S$/B | $C_2H_4$/B |
| 21 July-02 | S | 1.01 | 0.502 | 0.501 | 0.504 | | |
| | M | 2.19<br>0.941<br>0.940 | 0.849<br>0.538<br>0.539 | 0.495<br>0.509<br>0.521 | 0.851<br>0.720<br>0.863 | | |
| 21 July-02 | S | 100 | | | | | |
| | M | 98.7<br>99.9<br>100 | | | | | |
| 21 July-02 | S | | | 100 | | | |
| | M | | | 103<br>105<br>105 | | | |
| 29 July-02 | S | | 100 | | | | |
| | M | | 90.2<br>94.0<br>93.4 | | | | |
| 19 Sep.-02 | S | | | | | | 100 |
| | M | | | | | | 92.0<br>98.6<br>98.2 |
| 21 July-02 | S | | | | 100 | | |
| | M | | | | 103<br>103<br>103 | | |
| 21 July-02 | S | 54.0 | 4.01 | 20 | 20 | | |
| | M | 56.9<br>56.6<br>56.6 | 4.50<br>4.11<br>4.10 | 18.3<br>18.8<br>18.8 | 21.4<br>21.7<br>21.4 | | |
| 7 April-03 | S | 54.0 | 4.01 | 20 | 20 | | |
| | M | 58.9<br>58.7<br>58.7 | 5.10<br>4.89<br>4.92 | 18.0<br>18.3<br>18.2 | 19.7<br>19.8<br>20.5 | | |
| 29 July-02 | S | | | 45.1 | 54.9 | | |
| | M | | | 43.2<br>43.7<br>43.8 | 47.1<br>47.6<br>47.5 | | |

1). S=standard concentration.

2). M=measured concentration of standard gas, with triplicate analyses.

**Table A-3. The column notations.**

| System | Number of columns | Column notation |
|---|---|---|
| 1 (Cap water/MFT/CT) | 20 | C1-1, … C1-20 |
| 2 (Cap water/MFT/CT) | 6 | C2-1, …C2-6 |
| 3 (Cap water/MFT) | 12 | C3-1, …C3-12 |
| 4 (Cap water/CT) | 12 | C4-1, …C4-12 |

**Table A-4. The notations for samples in Systems 1, 3, and 4 (using System 1 as illustration).**

| Sample type | | Sample notation |
|---|---|---|
| Tedlar bag gas | | "C1-1-TGas", … "C1-20-TGas" |
| Cap water | | "C1-1-CapW", … "C1-20-CapW" |
| MFT | For MPN samples | "C1-1-MFT-U, C1-1-MFT-L", … "C1-20-MFT-U, C1-20-MFT-L" |
| | For other samples | "C1-1-MFT-U, C1-1-MFT-L", …"C1-20-MFT-U, C1-20-MFT-L" |
| CT | For MPN samples | "C1-1-CT-M", …."C1-20-CT-M" |
| | For other samples | "C1-1-CT-U, C1-1-CT-L", …."C1-20-CT-U, C1-20-CT-L" |

1). In case of more than one time Tedlar bag samplings from the same column, using suffix (1), (2), … to indicate the sequence of samplings from the same column. For example, "C1-1-TGas (1)", "C1-1-TGas (2)", "C1-1-TGas (3)",….

2). MFT-U and MFT-L are the upper and lower half zones of MFT in the MFT layer; while CT-U and CT-L are the upper and lower half zones of CT in the CT layer.

3). "M" means the CT sample for MPN analysis is taken from the middle of the CT layer.

**Table A-5. The notations for samples in System 2.**

| Sample type | Sample notation |
|---|---|
| Regular in-situ headspace gas analysis | "C2-1-OnGas (1), C2-1-OnGas (2), C2-1-OnGas (3), ...", ... "C2-6-OnGas (1), C2-6-OnGas (2), C2-6-OnGas (3), ..." |
| Regular in-situ cap water sampling | "C2-1-CapW (1), C2-1- CapW (2), C2-1- CapW (3), ...", ... "C2-6- CapW (1), C2-6- CapW (2), C2-6- CapW (3), ..." |
| MFT at column sacrificing | "C2-1-MFT-U, C2-1-MFT-L", ... "C2-6-MFT-U, C2-6-MFT-L" |
| CT at column sacrificing | "C2-1-CT-U, C2-1-CT-L", ..."C2-6-CT-U, C2-6-CT-L" |

1). The suffix (1), (2), (3), ... means the sequence of sampling times for the same column.
2). MFT-U and MFT-L are the upper and lower half zones of MFT in the MFT layer; while CT-U and CT-L are the upper and lower half zones of CT in the CT layer.

**Table A-6. Column fillings and incubation starting times in Systems 1, 3, and 4.**

| System | Column | Time of filling with MFT | Time of filling with CT |
|---|---|---|---|
| 1 (Cap water/MFT/CT) | C1-1 | From 2.0 pm to 3.0 pm, 4 Nov.-02 | 11.30 am, 5 Nov.-02 |
| | C1-2 | | 12.0 noon |
| | C1-3 | | 12.0 noon |
| | C1-4 | | 12.30 pm |
| | C1-5 | | 12.30 pm |
| | C1-6 | | 1.30 pm |
| | C1-8 | | 2.0 pm |
| | C1-9 | | 2.30 pm |
| | C1-10 | | 2.30 pm |
| | C1-11 | | 2.45 pm |
| | C1-12 | | 3.30 pm |
| | C1-13 | | 4.0 pm |
| | C1-14 | | 4.0 pm |
| | C1-15 | | 4.30 pm |
| | C1-16 | | 4.30 pm |
| | C1-17 | | 5.0 pm |
| | C1-18 | | 5.0 pm |
| | C1-19 | | 5.30 pm |
| | C1-20 | | 6.0 pm, on 5 Nov.-02 |
| 3 (Cap water/MFT) | C3-1, ...C3-12 | 4.30 pm, 6 Nov.-02 | |
| 4 (Cap water/CT) | C4-1, ...C4-12 | | 3.30 pm, 6 Nov.-02 |

1). For Systems 1 and 4, the time of filling with CT is regarded as the column setup and incubation starting time.
2). For Systems 3, the time of filling with MFT is regarded as the column setup and incubation starting time.
3). Column C1-7 was broken and discarded at column filling.

**Table A-7. Column filling and incubation starting time in System 2.**

| System | Column | Time of filling with MFT | Time of filling with CT |
|---|---|---|---|
| 2 (Cap water/MFT/CT) | C2-1 | 11.0 am, 9 Dec.-02 | 11.30 am, 10 Dec.-02 |
| | C2-2 | | 11.30 am |
| | C2-3 | | 11.30 am |
| | C2-4 | | 2.0 pm |
| | C2-5 | | 2.0 pm |
| | C2-6 | | 2.30 pm, 10 Dec.-02 |

1). The time of filling with CT is regarded as the column setup and incubation starting time.

# Appendix B. Results

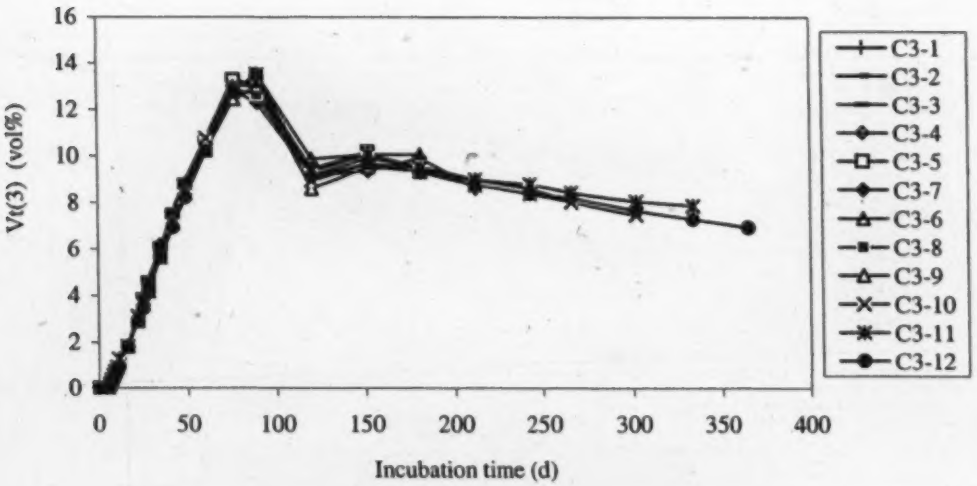



**Figure B-1. Total volume changes (Vt) in System 3.**

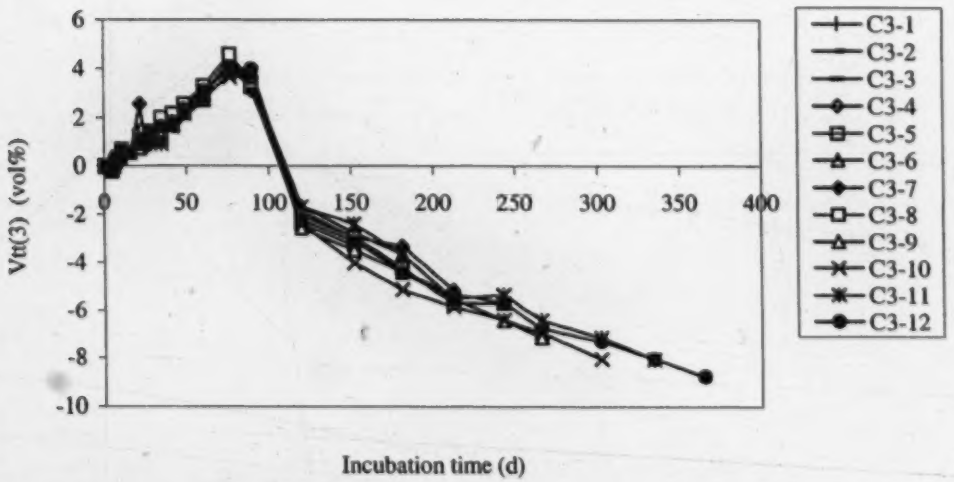



**Figure B-2. Total tailings volume changes (Vtt) in System 3.**

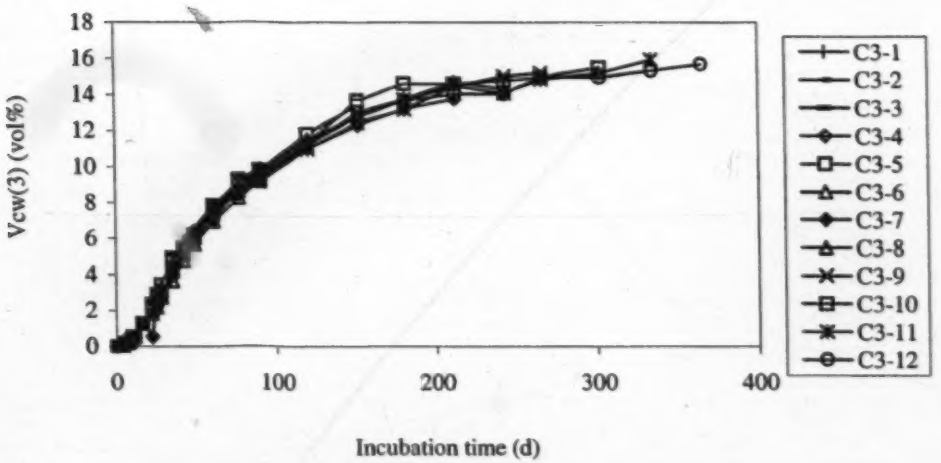



**Figure B-3. Cap water volumes (Vcw) in System 3.**

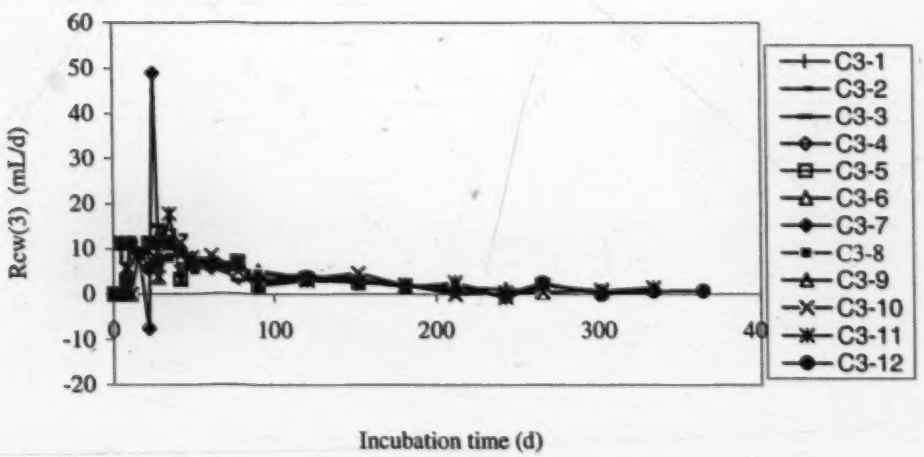



**Figure B-4. Cap water producing rates (Rcw) in System 3.**

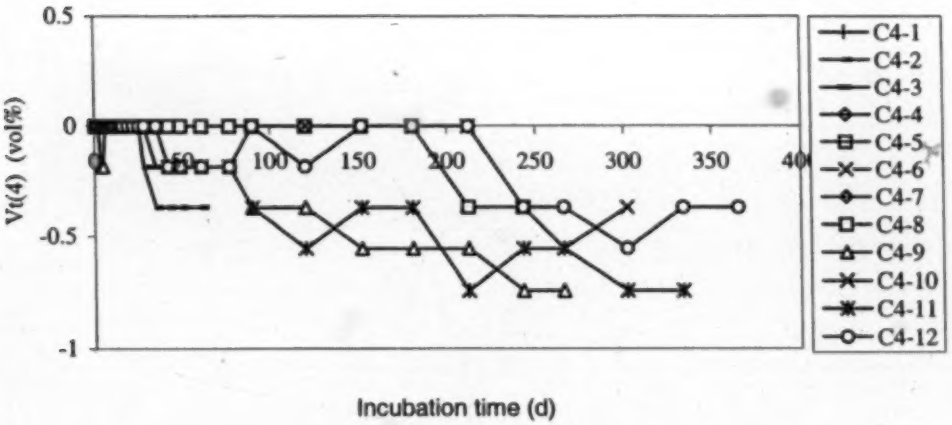



**Figure B-5. Total volume changes (Vt) in System 4.**

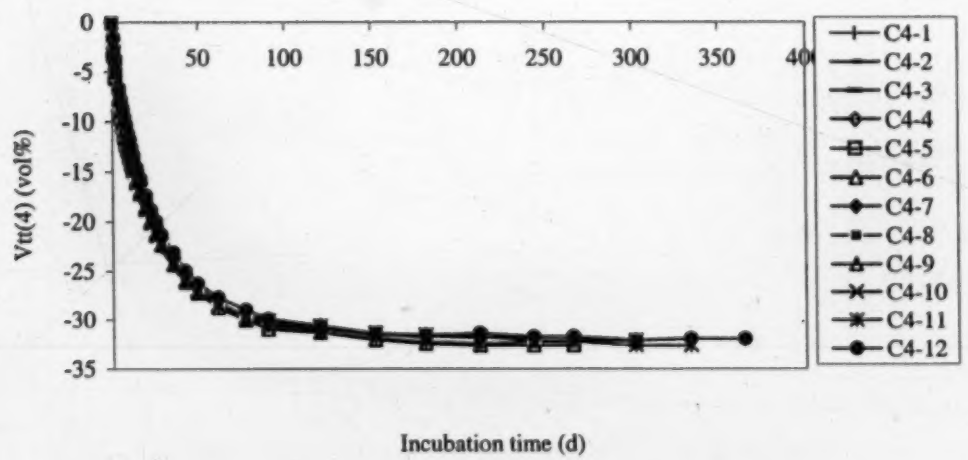



**Figure B-6. Total tailings volume changes (Vtt) in System 4.**

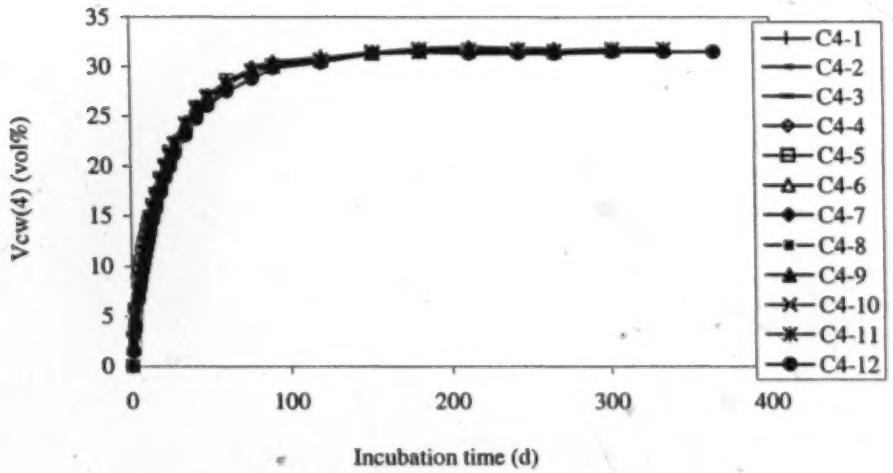



**Figure B-7. Cap water volumes (Vcw) in System 4.**

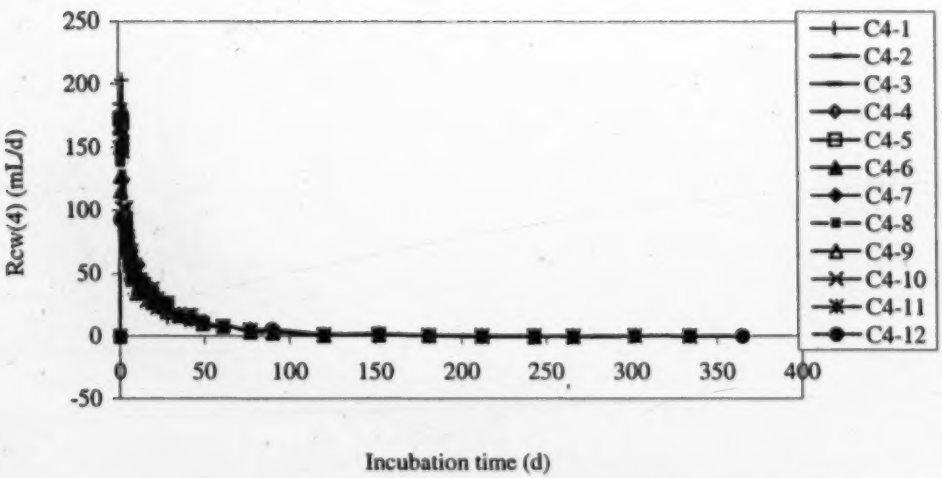



**Figure B-8. Cap water producing rates (Rcw) in System 4.**

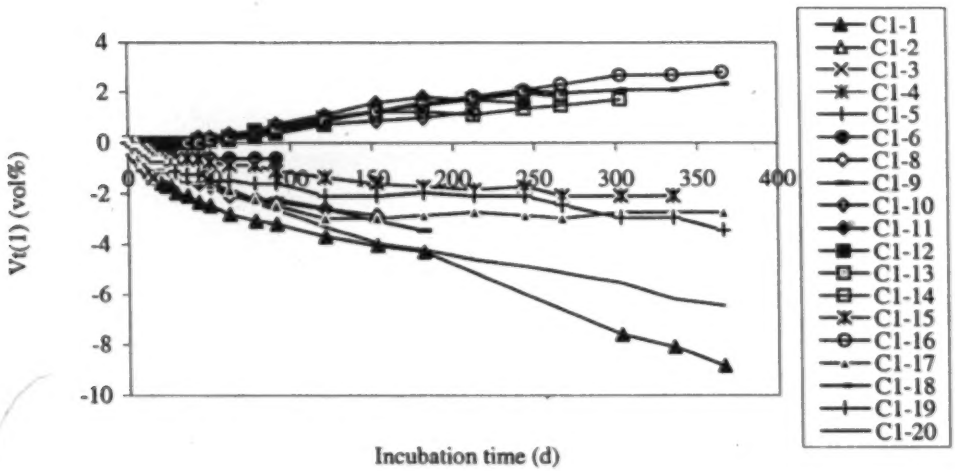



**Figure B-9. Total volume changes (Vt) in System 1.**

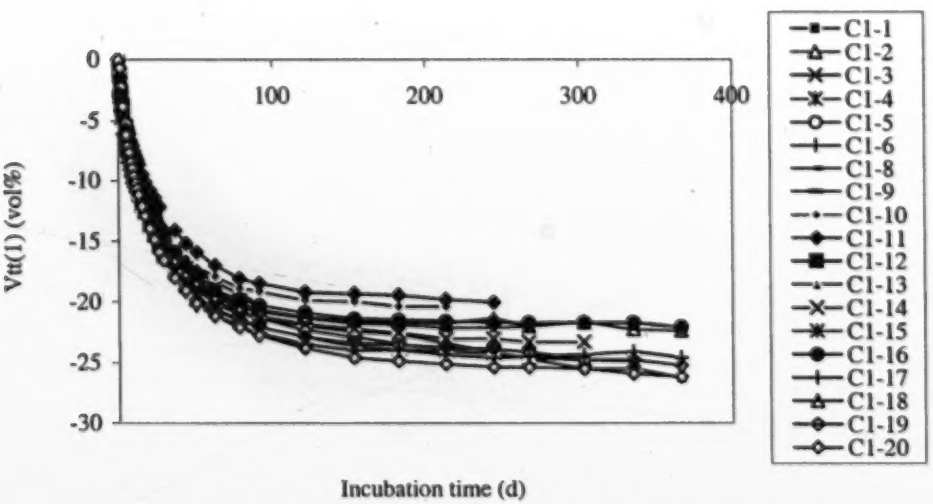



**Figure B-10. Total tailings volume changes (Vtt) in System 1.**

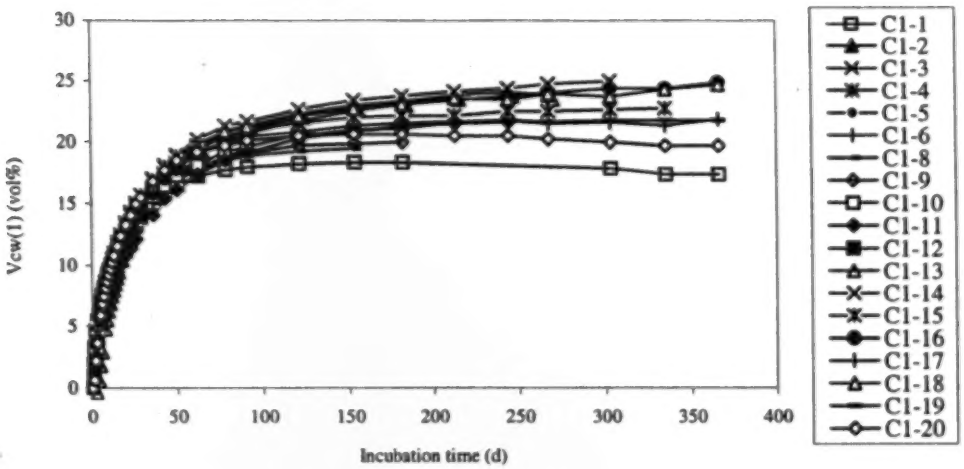



**Figure B-11. Cap water volumes (Vcw) in System 1.**

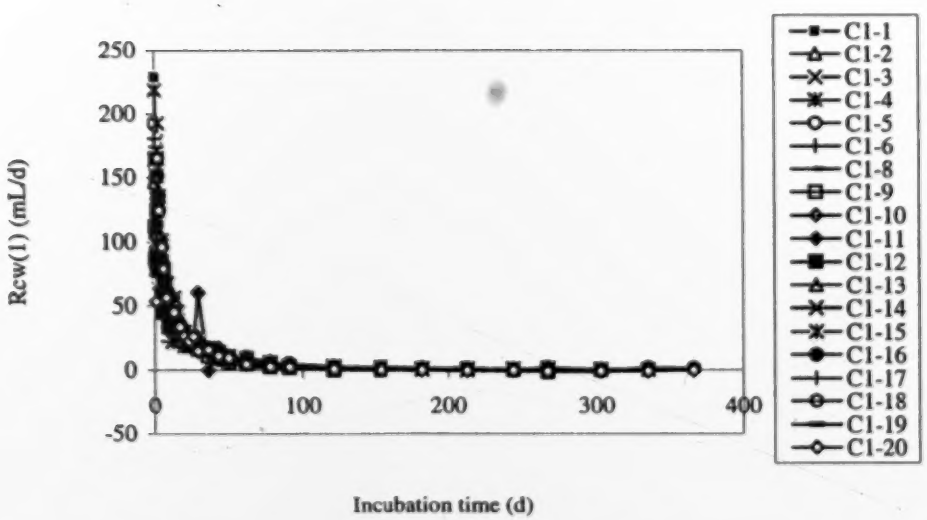



**Figure B-12. Cap water producing rates (Rcw) in System 1.**

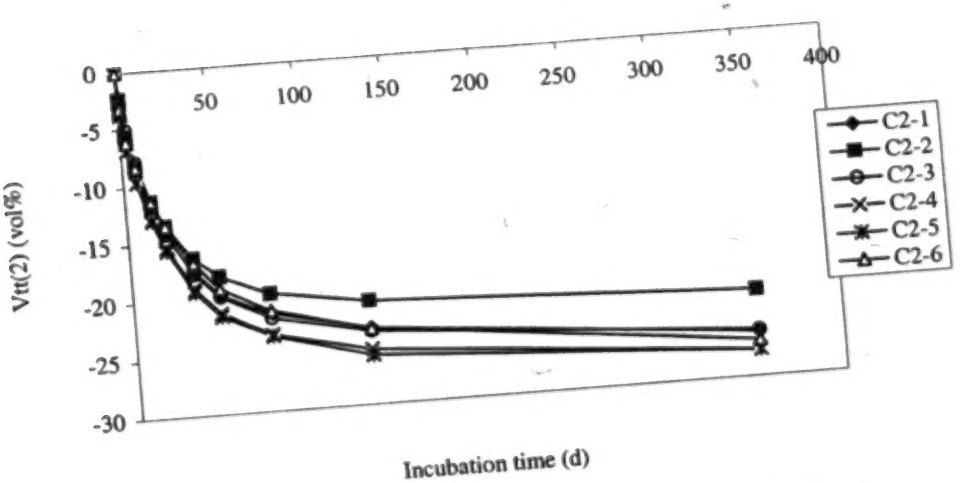



**Figure B-13. Total tailings volume changes (Vtt) in System 2.**

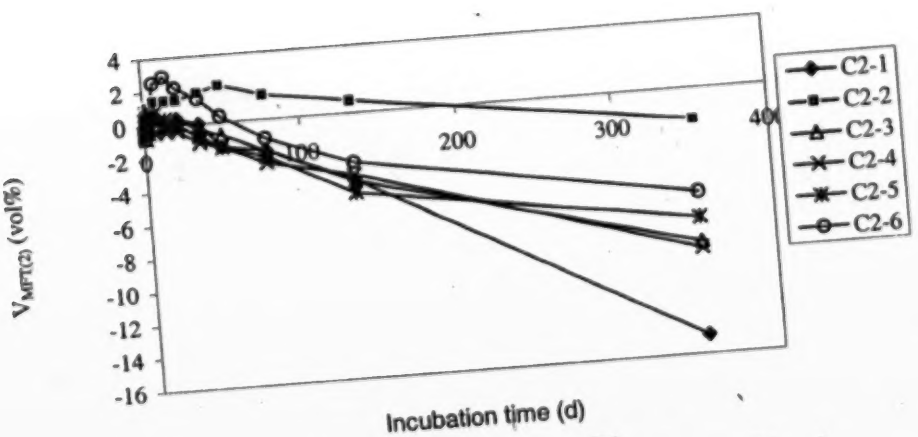



**Figure B-14. Individual MFT tailings volume changes ($V_{MFT}$) in System 2.**

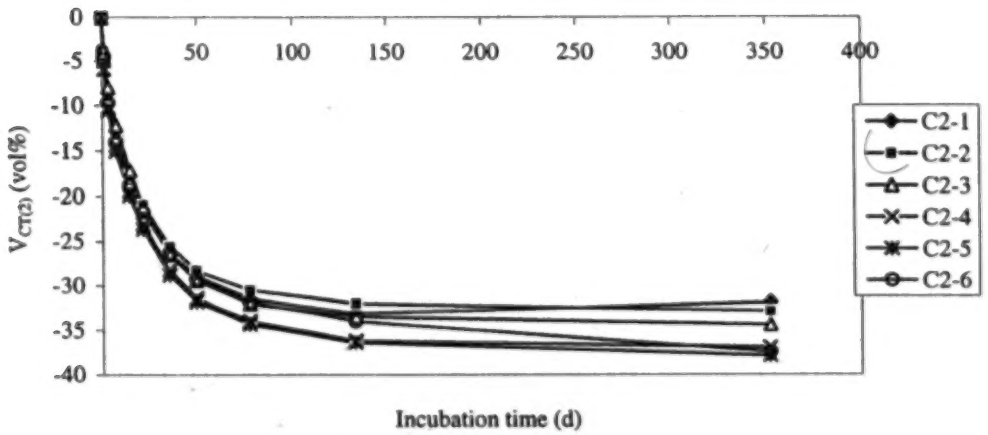



**Figure B-15. Individual CT tailings volume changes ($V_{CT}$) in System 2.**

**Table B-1. Absolute heights of cap water and MFT in System 3 over 1 y of incubation.**

| Column | $t_{incu}$ (d) | Height of MFT at time 0 (cm) | Height of cap water at column sacrificing (cm) | Height of MFT at column sacrificing (cm) |
|---|---|---|---|---|
| C3-1 | 28 | 54.5 | 1.7 | 55.2 |
| C3-2 | 61 | 54.7 | 4.3 | 56.2 |
| C3-3 | 90 | 54.7 | 5.1 | 56.7 |
| C3-4 | 120 | 54.9 | 6.2 | 53.8 |
| C3-5 | 152 | 55.0 | 7.4 | 53.2 |
| C3-6 | 181 | 54.7 | 7.5 | 52.7 |
| C3-7 | 212 | 54.5 | 7.5 | 51.7 |
| C3-8 | 243 | 54.5 | 7.8 | 51.4 |
| C3-9 | 266 | 54.6 | 8.3 | 50.7 |
| C3-10 | 302 | 54.8 | 8.5 | 50.4 |
| C3-11 | 334 | 54.6 | 8.7 | 50.2 |
| C3-12 | 365 | 54.8 | 8.6 | 50.0 |

Table B-2. Absolute heights of cap water and CT in System 4 over 1 y of incubation.

| Column | $t_{incu}$ (d) | Height of CT at time 0 (cm) | Height of cap water at column sacrificing (cm) | Height of CT at column sacrificing (cm) |
|---|---|---|---|---|
| C4-1 | 28 | 54.3 | 12.2 | 42.1 |
| C4-2 | 61 | 54.2 | 15.1 | 38.9 |
| C4-3 | 90 | 54.1 | 16.5 | 37.6 |
| C4-4 | 120 | 54.2 | 16.8 | 37.4 |
| C4-5 | 152 | 54.2 | 17.0 | 37.2 |
| C4-6 | 181 | 53.8 | 17.1 | 36.7 |
| C4-7 | 212 | 54.0 | 17.2 | 36.8 |
| C4-8 | 243 | 54.1 | 17.0 | 36.9 |
| C4-9 | 266 | 54.0 | 17.2 | 36.4 |
| C4-10 | 302 | 54.2 | 17.2 | 36.8 |
| C4-11 | 334 | 54.0 | 17.2 | 36.4 |
| C4-12 | 365 | 54.2 | 17.1 | 36.9 |

Table B-3. Absolute heights of cap water, MFT, and CT in System 1 over 1 y of incubation.

| | | Heights at time 0 (cm) | | Heights at column sacrificing (cm) | | |
|---|---|---|---|---|---|---|
| Column | $t_{incu}$ (d) | MFT | CT | Cap water | MFT | CT |
| C1-2 | 3 | 27.3 | 54.6 | 3.8 | 27.4 | 50.6 |
| C1-3 | 15 | 26.7 | 54.4 | 8.5 | 27.6 | 43.9 |
| C1-4 | 29 | 27.3 | 54.2 | 12.1 | 27.4 | 41.5 |
| C1-5 | 62 | 27.1 | 54.2 | 15.1 | 28.3 | 38.1 |
| C1-6 | 91 | 27.0 | 54.6 | 15.9 | 26.8 | 38.4 |
| C1-12 | 121 | 26.9 | 54.1 | 17.8 | 26.3 | 37.5 |
| C1-8 | 153 | 27.0 | 54.0 | 15.8 | 26.8 | 36.1 |
| C1-9 | 182 | 27.0 | 54.6 | 16.3 | 25.7 | 36.8 |
| C1-10 | 213 | 27.1 | 54.5 | 17.6 | 28.0 | 37.0 |
| C1-11 | 244 | 27.4 | 55.0 | 17.8 | 27.4 | 38.5 |
| C1-13 | 267 | 27.1 | 54.3 | 19.4 | 26.9 | 36.6 |
| C1-14 | 303 | 27.1 | NA | 20.4 | NA | NA |
| C1-15 | 335 | 27.2 | 54.5 | 18.6 | 25.9 | 35.5 |
| C1-16 | 366 | 27.2 | 54.8 | 20.4 | 27.6 | 36.3 |

1). NA=not available.

**Table B-4. Tedlar bag gas analyses for the major sacrificed columns in Systems 1, 3, and 4.**

| Sample | $t_{incu}$ (d) | Tedlar bag gas volume (mL) | GC analysis (vol%) | | | | |
|---|---|---|---|---|---|---|---|
| | | | $CH_4$ | $N_2$ | $O_2$ | $CO_2$ | Other gases |
| **System 1 (Cap water/MFT/CT)** | | | | | | | |
| C1-4-TGas | 29 | 45 | 0.044 | 77.8 | 20.7 | 0.636 | $H_2S$=0.605 |
| C1-5-TGas | 62 | 25 | 0.476 | 80.4 | 18.1 | 1.29 | |
| C1-6-TGas | 91 | 20 | 3.66 | 89.4 | 4.58 | 5.23 | |
| C1-12-TGas | 121 | 25 | 2.93 | 89.9 | 5.22 | 4.03 | |
| C1-8-TGas | 153 | 15 | 0.132 | 78.4 | 20.0 | 2.10 | $C_2H_4$=0.300; $H_2S$=0.429 |
| C1-9-TGas | 182 | 12 | 4.05 | 92.9 | 3.06 | 7.82 | $H_2S$=0.010 |
| C1-10-TGas | 213 | 20 | 4.57 | 90.5 | 4.45 | 3.88 | |
| C1-11-TGas | 244 | 10 | 0.662 | 80.7 | 17.0 | 1.96 | $H_2S$=1.25 |
| C1-13-TGas | 267 | 47 | 0.168 | 78.5 | 19.1 | 1.78 | |
| C1-14-TGas | 304 | 17 | 0.858 | 84.5 | 11.9 | 4.50 | |
| C1-15-TGas | 335 | 60 | 0.049 | 78.8 | 20.5 | 0.501 | |
| C1-16-TGas | 366 | 45 | 0.033 | 82.4 | 22.0 | 0.241 | |
| **System 3 (Cap water/MFT)** | | | | | | | |
| C3-1-TGas | 28 | 325 | 0.393 | 95.4 | 3.19 | 1.43 | |
| C3-2-TGas | 61 | 430 | 0.443 | 85.0 | 13.5 | 1.77 | |
| C3-3-TGas | 90 | 350 | 4.72 | 87.5 | 6.36 | 3.06 | |
| C3-4-TGas (1) | 90 | 1020 | 3.65 | 93.0 | 1.69 | 3.60 | |
| C3-4-TGas (2) | 120 | 400 | 11.6 | 79.4 | 8.00 | 3.76 | |
| C3-5-TGas | 152 | 45 | 0.336 | 77.8 | 20.8 | 0.348 | $C_2H_4$=0.115; $H_2S$=0.932 |
| C3-6-TGas | 181 | 440 | 20.9 | 74.3 | 3.88 | 5.29 | |
| C3-7-TGas | 212 | 485 | 20.7 | 70.60 | 6.82 | 5.880 | |
| C3-8-TGas | 243 | 20 | 0.846 | 77.0 | 20.3 | 0.818 | $C_2H_4$=0.137; $H_2S$=0.058 |
| C3-9-TGas | 266 | 74 | 0.557 | 77.4 | 20.5 | 0.559 | |
| C3-10-TGas | 303 | 460 | 18.0 | 69.4 | 10.2 | 5.45 | |
| C3-11-TGas (1) | 90 | 1030 | 3.53 | 93.1 | 1.59 | 3.47 | |
| C3-11-TGas (2) | 334 | 1000 | 34.0 | 61.3 | 2.08 | 6.82 | |
| C3-12-TGas | 365 | 171 | 7.09 | 75.3 | 17.3 | 3.63 | |

**Table B-4.** (*continued*).

| Sample | $t_{incu}$ (d) | Tedlar bag gas volume (mL) | GC analysis (vol%) | | | | |
|---|---|---|---|---|---|---|---|
| | | | $CH_4$ | $N_2$ | $O_2$ | $CO_2$ | Other gases |
| **System 4 (Cap water/CT)** | | | | | | | |
| C4-1-TGas | 28 | 80 | 0.040 | 93.7 | 5.54 | 0.885 | |
| C4-2-TGas | 61 | 30 | 0.061 | 91.3 | 8.76 | 0.646 | $C_2H_4$=0.009 $H_2S$=0.216 |
| C4-3-TGas | 90 | 60 | 0.085 | 95.0 | 4.76 | 1.41 | |
| C4-4-TGas | 120 | 40 | nd | 80.5 | 17.4 | 1.85 | |
| C4-5-TGas | 152 | 180 | 0.109 | 93.3 | 4.28 | 2.65 | $H_2S$=0.440 |
| C4-6-TGas | 181 | 30 | 0.053 | 85.7 | 12.2 | 4.42 | |
| C4-7-TGas | 212 | 17 | 0.291 | 83.0 | 15.5 | 0.971 | $C_2H_4$=0.170; $H_2S$=0.138 |
| C4-8-TGas | 243 | 30 | 0.129 | 95.4 | 3.30 | 1.19 | |
| C4-9-TGas | 266 | 42 | nd | 77.8 | 20.9 | 0.141 | |
| C4-10-TGas | 303 | 84 | 0.140 | 94.7 | 3.00 | 3.26 | |
| C4-11-TGas | 334 | 155 | 0.044 | 80.0 | 17.2 | 2.74 | |
| C4-12-TGas | 365 | 308 | 0.071 | 83.4 | 15.7 | 2.81 | |

1). nd=non-detectable.

**Table B-5. The regular in-situ headspace gas analyses for the columns in System 2.**

| Sample | $t_{incu}$ (d) | GC analysis (vol%) | | | | |
|---|---|---|---|---|---|---|
| | | $CH_4$ | $N_2$ | $O_2$ | $CO_2$ | Other gases |
| C2-1-OnGas (1) | 1 | 0.045 | 83.3 | 16.3 | 0.347 | $C_2H_4$=0.092 |
| C2-2-OnGas (1) | 1 | 0.070 | 83.5 | 15.8 | 0.658 | $C_2H_4$=0.278 |
| C2-3-OnGas (1) | 1 | 0.077 | 85.3 | 14.1 | 0.553 | $C_2H_4$=0.024 |
| C2-4-OnGas (1) | 1 | 0.044 | 82.3 | 16.8 | 0.399 | $C_2H_4$=0.007 |
| C2-5-OnGas (1) | 1 | 0.1005 | 86.2 | 13.0 | 0.526 | $C_2H_4$=0.236 |
| C2-6-OnGas (1) | 1 | 0.120 | 87.4 | 11.7 | 0.010 | $C_2H_4$=0.012 |
| C2-1-OnGas (2) | 3 | 0.145 | 99.0 | 13.4 | 0.805 | |
| C2-2-OnGas (2) | 3 | 0.098 | 82.6 | 17.3 | 0.011 | $C_2H_4$=0.020 |
| C2-3-OnGas (2) | 3 | 0.130 | 85.8 | 14.0 | 0.006 | $C_2H_4$=0.072 |
| C2-4-OnGas (2) | 3 | 0.146 | 86.3 | 13.2 | 0.743 | $C_2H_4$=0.013 |
| C2-5-OnGas (2) | 3 | 0.100 | 84.9 | 14.7 | 0.563 | $C_2H_4$=0.016 |
| C2-6-OnGas (2) | 3 | 0.145 | 87.2 | 12.1 | 0.770 | $C_2H_4$=0.022 |
| C2-1-OnGas (3) | 7 | 0.186 | 83.6 | 17.8 | 1.060 | $H_2S$=0.612 |
| C2-2-OnGas (3) | 7 | 0.095 | 79.7 | 20.7 | 0.699 | $C_2H_4$=0.036 |
| C2-3-OnGas (3) | 7 | 0.203 | 83.1 | 16.8 | 0.936 | |
| C2-4-OnGas (3) | 7 | 0.234 | 83.7 | 15.9 | 1.06 | $C_2H_4$=0.012 |
| C2-5-OnGas (3) | 7 | 0.130 | 81.3 | 18.3 | 0.761 | |
| C2-6-OnGas (3) | 7 | 0.031 | 79.3 | 20.6 | 0.577 | $C_2H_4$=0.031 |
| C2-1-OnGas (4) | 14 | 0.298 | 83.2 | 18.5 | 1.71 | $H_2S$=0.006 |
| C2-2-OnGas (4) | 14 | 0.109 | 78.9 | 20.8 | 0.860 | |
| C2-3-OnGas (4) | 14 | 0.382 | 81.9 | 16.6 | 1.80 | |
| C2-4-OnGas (4) | 14 | 0.370 | 81.8 | 16.3 | 1.87 | |
| C2-5-OnGas (4) | 14 | 0.304 | 80.6 | 17.7 | 1.57 | |
| C2-6-OnGas (4) | 14 | 0.573 | 83.0 | 14.7 | 2.03 | |
| C2-1-OnGas (5) | 21 | 0.129 | 87.5 | 14.2 | 1.75 | |
| C2-2-OnGas (5) | 21 | 0.072 | 80.3 | 19.7 | 0.647 | |
| C2-3-OnGas (5) | 21 | 0.166 | 87.8 | 11.7 | 1.12 | |
| C2-4-OnGas (5) | 21 | 0.165 | 88.8 | 10.5 | 1.13 | |
| C2-5-OnGas (5) | 21 | 0.142 | 86.1 | 12.9 | 0.963 | |
| C2-6-OnGas (5) | 21 | 0.263 | 89.4 | 9.58 | 1.23 | |

**Table B-5.** *(continued).*

| Sample | $t_{incu}$ (d) | Gas GC analysis (vol%) | | | | |
|---|---|---|---|---|---|---|
| | | $CH_4$ | $N_2$ | $O_2$ | $CO_2$ | Other gases |
| C2-1-OnGas (6) | 35 | 0.151 | 80.1 | 19.2 | 2.40 | |
| C2-2-OnGas (6) | 35 | 0.054 | 77.8 | 20.7 | 0.625 | $C_2H_4$=0.033 |
| C2-3-OnGas (6) | 35 | 0.219 | 80.9 | 17.3 | 1.52 | $C_2H_4$=0.010 |
| C2-4-OnGas (6) | 35 | 0.236 | 81.4 | 16.3 | 1.79 | |
| C2-5-OnGas (6) | 35 | 0.181 | 80.0 | 18.0 | 1.29 | |
| C2-6-OnGas (6) | 35 | 0.365 | 82.8 | 15.0 | 1.77 | |
| C2-1-OnGas (7) | 49 | 0.107 | 79.5 | 19.8 | 1.09 | |
| C2-2-OnGas (7) | 49 | 0.045 | 78.1 | 20.6 | 0.621 | $C_2H_4$=0.110 |
| C2-3-OnGas (7) | 49 | 0.195 | 80.8 | 18.9 | 1.90 | $H_2S$=0.009 |
| C2-4-OnGas (7) | 49 | 0.218 | 79.8 | 17.8 | 1.68 | |
| C2-5-OnGas (7) | 49 | 0.147 | 79.0 | 18.9 | 1.31 | $C_2H_4$=0.0079 |
| C2-6-OnGas (7) | 49 | 0.338 | 80.9 | 16.7 | 1.85 | $H_2S$=0.021 |
| C2-1-OnGas (8) | 77 | 0.068 | 79.2 | 20.2 | 1.15 | |
| C2-2-OnGas (8) | 77 | 0.038 | 78.2 | 20.8 | 0.763 | $C_2H_4$=0.093 $H_2S$=0.057 |
| C2-3-OnGas (8) | 77 | 0.137 | 78.6 | 19.4 | 1.53 | |
| C2-4-OnGas (8) | 77 | 0.158 | 78.9 | 18.9 | 1.67 | |
| C2-5-OnGas (8) | 77 | 0.075 | 78.5 | 19.6 | 1.26 | |
| C2-6-OnGas (8) | 77 | 0.254 | 79.7 | 18.0 | 1.72 | |
| C2-1-OnGas (9) | 133 | 0.047 | 81.9 | 20.9 | 1.19 | $C_2H_4$=0.203 |
| C2-2-OnGas (9) | 133 | 0.030 | 79.4 | 21.0 | 0.685 | $C_2H_4$=0.086; $H_2S$=0.070. |
| C2-3-OnGas (9) | 133 | 0.095 | 79.2 | 19.5 | 1.688 | |
| C2-4-OnGas (9) | 133 | 0.100 | 79.0 | 19.1 | 1.84 | |
| C2-5-OnGas (9) | 133 | 0.061 | 78.8 | 19.7 | 1.30 | |
| C2-6-OnGas (9) | 133 | 0.156 | 79.6 | 18.1 | 2.29 | |
| C2-1-OnGas (10) | 352 | nd | 77.2 | 20.8 | 0.149 | |
| C2-4-OnGas (10) | 352 | nd | 76.9 | 20.6 | 0.275 | |
| C2-6-OnGas (10) | 352 | 0.047 | 77.4 | 19.4 | 1.19 | |

1). nd=non-detectable.

**Table B-6. Physical and chemical analyses in baseline MFT and CT.**

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| Base MFT 1 | 0 | 7.96 | 4430 | BDL | 9 | BDL | 1030 | 16 | 15.5 | 22.4 | BDL | 860 | BDL | 1491 | BDL | 3.9 | 0.58 | BDL | 4.9 | 0.953 |
| Base MFT 2 | 0 | 7.96 | 4460 | BDL | 10.4 | BDL | 1020 | 17 | 15.7 | 23.1 | BDL | 890 | 13.9 | 1522 | BDL | 4 | 0.6 | BDL | 5.08 | 0.973 |
| Base MFT 3 | 0 | 7.9 | 4600 | BDL | 10.1 | BDL | 1030 | 15.6 | 16.2 | 23.8 | BDL | 850 | 22.6 | 1350 | BDL | 3.50 | 0.61 | BDL | 4.68 | 0.96 |
| Base CT 1 | 0 | 8.22 | 5770 | 270 | 7.5 | 0.4 | 1240 | 19 | 24.8 | 68.7 | BDL | 1000 | 724 | 739 | BDL | 3 | 0.2 | BDL | 3.87 | 1.48 |
| Base CT 2 | 0 | 8.17 | 6120 | 260 | 7.9 | 4 | 1310 | 20 | 26.4 | 76 | BDL | 1100 | 897 | 806 | BDL | 3.1 | 0.13 | BDL | 3.91 | 1.61 |
| Base CT 3 | 0 | 8.08 | 6260 | 260 | 8.4 | 10 | 1400 | 20 | 29.5 | 82 | BDL | 1000 | 1100 | 731 | BDL | 2.9 | 0.2 | BDL | 3.9 | 1.81 |
| Base CT 4 | 0 | 8.0 | 6350 | 300 | 8.4 | 3 | 1370 | 19.0 | 27.7 | 77.7 | BDL | 1100 | 1070 | 674 | BDL | 2.60 | 0.09 | BDL | 3.50 | 1.58 |
| Base CT 5 | 0 | 8.1 | 6770 | 280 | 9.0 | 3 | 1470 | 19.2 | 29.9 | 98.6 | BDL | 1100 | 1350 | 642 | BDL | 2.55 | 0.05 | BDL | 3.29 | 1.75 |
| Base CT 6 | 0 | 8.0 | 7900 | 280 | 7.9 | 3 | 1810 | 20.9 | 33.9 | 114.0 | 46.0 | 1100 | 1660 | 1160 | BDL | 2.69 | 0.08 | BDL | 3.47 | 2.06 |

**Table B-6.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | AVS (mg/kg of dry sample) | Particle Size Distribution (% of solids less than) | | | | | |
| | | | | | | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| Base MFT 1 | 0 | 37.92 | 3.2 | -9 | 73.6 | 99.8 | 95 | 80 | 57.4 | 36.2 | 12.7 |
| Base MFT 2 | 0 | 38.03 | 2.94 | 2 | NA | 92 | 78.9 | 62.3 | 44.7 | 28.3 | 9.94 |
| Base MFT 3 | 0 | 38.63 | 3.47 | -3 | NA | 86.3 | 73.4 | 57.4 | 41.1 | 26.3 | 9.73 |
| Base CT 1 | 0 | 57.19 | 0.81 | -202 | NA | NA | NA | NA | NA | NA | NA |
| Base CT 2 | 0 | 56.49 | 0.76 | -202 | 80 | 34.3 | 29.3 | 23.8 | 16.8 | 9.73 | 3.26 |
| Base CT 3 | 0 | 56.58 | 1.13 | -196 | NA | 38.2 | 31.9 | 25.2 | 17.8 | 10.6 | 3.59 |
| Base CT 4 | 0 | 59.02 | 1.27 | -188 | NA | 34.2 | 27.9 | 21.3 | 15.0 | 9.90 | 4.19 |
| Base CT 5 | 0 | 55.65 | 1.17 | -169 | NA | 37.7 | 30.9 | 23.7 | 16.7 | 10.90 | 4.53 |
| Base CT 6 | 0 | 54.27 | 1.2 | -172 | NA | 34.4 | 28.0 | 21.5 | 15.2 | 10.00 | 4.21 |

1). NA=not available.
2). BDL=below detection limits.

**Table B-7. Physical and chemical analyses in System 3 .**

| | | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| Sample | $t_{incu}$ (d) | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C3-1-CapW | 28 | 8.2 | 4420 | BDL | NA | BDL | 950 | 15 | 13.2 | 19 | BDL | 1700 | 11.9 | 1491 | BDL | 3.5 | 0.4 | BDL | 4.06 | 0.79 |
| C3-1-MFT-U | 28 | 8.3 | 4280 | BDL | 8.7 | BDL | 939 | 15 | 12.8 | 17 | BDL | 840 | 17.3 | 1430 | BDL | 1.2 | 0.8 | BDL | 3.39 | 0.73 |
| C3-1-MFT-L | 28 | 8.1 | 4350 | BDL | 9.4 | BDL | 915 | 14 | 12.6 | 16 | BDL | 850 | 22.2 | 1388 | BDL | 0.9 | 0.4 | BDL | 3.06 | 0.71 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-2-CapW | 61 | 7.9 | 4540 | BDL | 8.8 | BDL | 1000 | 15 | 15.3 | 22 | BDL | 900 | 18.9 | NA | BDL | 2.7 | 0.5 | BDL | 5.12 | 0.93 |
| C3-2-MFT-U | 61 | 8.1 | 4300 | BDL | 9.5 | BDL | 941 | 14 | 11.8 | 16 | BDL | 830 | 9.36 | 1423 | BDL | 3 | 0.4 | BDL | 3.94 | 0.7 |
| C3-2-MFT-L | 61 | 8.2 | 4300 | BDL | 9.5 | BDL | 972 | 15 | 12.6 | 17 | BDL | 830 | BDL | 1429 | BDL | 3.1 | 0.4 | BDL | 4.28 | 0.76 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-3-CapW | 90 | 7.8 | 4340 | BDL | 9.0 | BDL | 906 | 14 | 14.4 | 20 | BDL | 850 | BDL | 1525 | BDL | 3.7 | 0.4 | BDL | 5.03 | 0.9 |
| C3-3-MFT-U | 90 | 8.1 | 4340 | BDL | 9.3 | BDL | 940 | 15 | 12.3 | 17 | BDL | 840 | BDL | 1490 | BDL | 3 | 0.4 | BDL | 3.77 | 0.74 |
| C3-3-MFT-L | 90 | 8 | 4310 | BDL | 9.3 | BDL | 943 | 15 | 11.8 | 16 | BDL | 840 | BDL | 1426 | BDL | 2.9 | 0.4 | BDL | 3.55 | 0.71 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-4-CapW | 120 | 7.8 | 4530 | BDL | 9.1 | BDL | 977 | 14 | 14.8 | 21 | BDL | 860 | BDL | 1547 | BDL | 3.9 | 0.4 | BDL | 5.26 | 0.91 |
| C3-4-MFT-U | 120 | 7.8 | 4390 | BDL | 9.1 | BDL | 971 | 14 | 13.8 | 19 | 6.1 | 830 | BDL | 1500 | BDL | 2.9 | 0.4 | BDL | 4.09 | 0.82 |
| C3-4-MFT-L | 120 | 7.8 | 4370 | BDL | 9.5 | BDL | 998 | 14 | 14.3 | 19 | 5.9 | 850 | BDL | 1497 | BDL | 2.9 | 0.4 | BDL | 4.11 | 0.84 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-5-CapW | 152 | NA | NA | NA | NA | BDL | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA |
| C3-5-MFT-U | 152 | 7.9 | 4120 | BDL | 8.7 | BDL | 881 | 13 | 12.6 | 18 | BDL | 820 | 4.68 | 1514 | BDL | 2.8 | 0.4 | BDL | 4.04 | 0.76 |
| C3-5-MFT-L | 152 | 8.1 | 3900 | BDL | 8.6 | BDL | 820 | 13 | 11.2 | 16 | BDL | 740 | 4.46 | 1446 | BDL | 2.7 | 0.4 | BDL | 3.69 | 0.67 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-6-CapW | 181 | 7.7 | 4460 | BDL | 9.7 | BDL | 1020 | 15 | 15.7 | 23 | BDL | 840 | BDL | 1482 | BDL | 4 | 0.4 | BDL | 5.64 | 0.99 |
| C3-6-MFT-U | 181 | 7.9 | 4370 | BDL | | BDL | 980 | 16 | 13.7 | 20 | BDL | 850 | BDL | 1540 | BDL | 3.3 | 0.4 | BDL | 4.55 | 0.86 |
| C3-6-MFT-L | 181 | 7.9 | 4340 | BDL | | BDL | 1040 | 17 | 14.6 | 21 | BDL | 800 | BDL | 1530 | BDL | 3.1 | 0.5 | BDL | 4.46 | 0.91 |

**Table B-7.** *(continued).*

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C3-7-CapW | 212 | 7.7 | 4520 | BDL | 11.7 | BDL | 1150 | 16 | 16 | 24 | BDL | 840 | BDL | 1580 | BDL | 4.5 | 0.5 | BDL | 5.92 | 1.03 |
| C3-7-MFT-U | 212 | 7.9 | 4320 | BDL | 11.3 | BDL | 1130 | 15 | 13 | 17 | BDL | 840 | BDL | 1390 | BDL | 3.3 | 0.4 | BDL | 4.21 | 0.78 |
| C3-7-MFT-L | 212 | 8 | 4220 | BDL | 11.1 | BDL | 1100 | 14 | 13 | 17 | BDL | 830 | BDL | 1390 | BDL | 3 | 0.4 | BDL | 3.96 | 0.76 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-8-CapW | 243 | 8.1 | 4520 | BDL | 9.2 | BDL | 1130 | 15 | 15 | 21 | BDL | 800 | BDL | 1550 | BDL | 4.1 | 0.5 | BDL | 4.85 | 0.96 |
| C3-8-MFT-U | 243 | 7.7 | 4350 | BDL | 9.7 | BDL | 1080 | 14 | 13 | 17 | BDL | 810 | BDL | 1400 | BDL | 3.1 | 0.4 | BDL | 4.06 | 0.78 |
| C3-8-MFT-L | 243 | 7.9 | 4280 | BDL | 10.0 | BDL | 1050 | 14 | 12 | 15 | BDL | 810 | BDL | 1350 | BDL | 2.7 | 0.4 | BDL | 3.47 | 0.71 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-9-CapW | 266 | 8 | 4530 | BDL | 7.9 | BDL | 1040 | 14 | 14 | 20 | BDL | 840 | 11.2 | 1590 | BDL | 3.7 | 0.5 | BDL | 4.42 | 0.86 |
| C3-9-MFT-U | 266 | 7.9 | 4330 | BDL | 10.2 | BDL | 1010 | 14 | 12 | 16 | BDL | 820 | BDL | 1460 | BDL | 3.1 | 0.3 | BDL | 4.15 | 0.72 |
| C3-9-MFT-L | 266 | 7.7 | 4280 | BDL | 10.8 | BDL | 1020 | 14 | 12 | 15 | BDL | 810 | BDL | 1400 | BDL | 3.1 | 0.3 | BDL | 4.02 | 0.7 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-10-CapW | 302 | 7.5 | 4670 | BDL | 9.4 | BDL | 1160 | 16 | 17 | 24 | BDL | 850 | 9.55 | 1600 | BDL | 4.4 | 0.6 | BDL | 6.08 | 1.06 |
| C3-10-MFT-U | 302 | 7.6 | 4500 | BDL | 9.5 | BDL | 1100 | 15 | 14 | 18 | BDL | 1700 | BDL | 1400 | BDL | 3.5 | 0.4 | BDL | 4.98 | 0.82 |
| C3-10-MFT-L | 302 | 7.7 | 4360 | BDL | NA | BDL | 1060 | 14 | 13 | 17 | BDL | 430 | 11.8 | 1390 | BDL | 3.2 | 0.4 | BDL | 4.44 | 0.76 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-11-CapW | 334 | 8.3 | 4570 | BDL | NA | BDL | 1090 | 17 | 18 | 27 | BDL | 850 | 11.3 | 1610 | BDL | 4.7 | 0.8 | BDL | 6.63 | 1.06 |
| C3-11-MFT-U | 334 | 7.9 | 4440 | BDL | NA | BDL | 1030 | 15 | 15 | 19 | BDL | 850 | BDL | 1490 | BDL | 3.3 | 0.4 | BDL | 4.36 | 0.85 |
| C3-11-MFT-L | 334 | 7.9 | 4310 | BDL | NA | BDL | 980 | 15 | 14 | 18 | BDL | 850 | BDL | 1420 | BDL | 3 | 0.4 | BDL | 4.13 | 0.79 |
| | | | | | | | | | | | | | | | | | | | | |
| C3-12-CapW | 365 | 7.8 | 4630 | BDL | 8.2 | BDL | 1150 | 16 | 17 | 24 | BDL | 850 | 10.3 | 1620 | BDL | 4.6 | 0.5 | BDL | 6.19 | 1.03 |
| C3-12-MFT-U | 365 | 7.8 | 4490 | BDL | 8.8 | BDL | 1110 | 16 | 14 | 18 | BDL | 860 | BDL | 1460 | BDL | 3.5 | 0.4 | BDL | 4.76 | 0.82 |
| C3-12-MFT-L | 365 | 7.8 | 4410 | BDL | 10.0 | BDL | 1090 | 16 | 14 | 19 | BDL | 840 | BDL | 1460 | BDL | 3.6 | 0.4 | BDL | 4.88 | 0.82 |

**Table B-7.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Particle Size Distribution (% of solids less than) | | | | | |
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| C3-1-CapW | 28 | | | | | | | | | |
| C3-1-MFT-U | 28 | 38.3 | 3.1 | 21 | 91.2 | 77.4 | 59.9 | 42.5 | 27.4 | 10.7 |
| C3-1-MFT-L | 28 | 39.0 | 3.37 | 33 | 89.6 | 76.7 | 60.1 | 43.1 | 27.3 | 9.9 |
| | | | | | | | | | | |
| C3-2-CapW | 61 | | | | | | | | | |
| C3-2-MFT-U | 61 | 39.7 | 3.21 | 40 | 95.7 | 83 | 66.7 | 48.6 | 31.2 | 11.8 |
| C3-2-MFT-L | 61 | 40.3 | 3.28 | 42 | 88.6 | 75.5 | 60.2 | 43.1 | 27.2 | 10.3 |
| | | | | | | | | | | |
| C3-3-CapW | 90 | | | | | | | | | |
| C3-3-MFT-U | 90 | 40.9 | 3.47 | 21 | 91.3 | 78.1 | 61.5 | 43.5 | 27.6 | 10.4 |
| C3-3-MFT-L | 90 | 41.2 | 3.48 | 53 | 95.2 | 81.9 | 65.2 | 47.1 | 30.6 | 12.2 |
| | | | | | | | | | | |
| C3-4-CapW | 120 | | | | | | | | | |
| C3-4-MFT-U | 120 | 41.8 | 3.51 | 31 | 95.5 | 82.2 | 65.3 | 46.9 | 30 | 11.8 |
| C3-4-MFT-L | 120 | 41.9 | 3.54 | 38 | 91.2 | 78.4 | 62.3 | 44.5 | 28 | 10.8 |
| | | | | | | | | | | |
| C3-5-CapW | 152 | | | | | | | | | |
| C3-5-MFT-U | 152 | 41.6 | 3.52 | 49 | 90.2 | 77.7 | 61.3 | 44.4 | 28.4 | 9.37 |
| C3-5-MFT-L | 152 | NA | NA | NA | NA | NA | NA | NA | NA | NA |
| | | | | | | | | | | |
| C3-6-CapW | 181 | | | | | | | | | |
| C3-6-MFT-U | 181 | 42.4 | 3.37 | 27 | 83 | 69.7 | 52.9 | 37 | 22.6 | 7.89 |
| C3-6-MFT-L | 181 | 43.2 | 3.65 | 39 | 85.9 | 73.1 | 57.2 | 40.2 | 24.9 | 8.79 |

**Table B-7.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | Particle Size Distribution (% of solids less than) | | | | | |
| | | | | | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| C3-7-CapW | 212 | | | | | | | | | |
| C3-7-MFT-U | 212 | 44.1 | 3.54 | 68 | 86.7 | 73.4 | 57.4 | 40 | 24.5 | 9.3 |
| C3-7-MFT-L | 212 | 44.3 | 3.55 | 37 | 91.2 | 79.4 | 64.1 | 46.6 | 29.5 | 11.2 |
| | | | | | | | | | | |
| C3-8-CapW | 243 | | | | | | | | | |
| C3-8-MFT-U | 243 | 42.5 | 3.67 | 33.2 | 95.7 | 83.2 | 66.9 | 49.4 | 32.3 | 12.8 |
| C3-8-MFT-L | 243 | 43.8 | 3.32 | 47.1 | 96 | 79.8 | 67.9 | 47.6 | 32.0 | 12.1 |
| | | | | | | | | | | |
| C3-9-CapW | 266 | | | | | | | | | |
| C3-9-MFT-U | 266 | 46.8 | 3.81 | 67.4 | 90.3 | 76.8 | 59.9 | 42.3 | 26.7 | 8.1 |
| C3-9-MFT-L | 266 | 43.0 | 3.31 | 66.6 | 92 | 79.6 | 63.6 | 45.6 | 28.3 | 8.1 |
| | | | | | | | | | | |
| C3-10-CapW | 302 | | | | | | | | | |
| C3-10-MFT-U | 302 | 42.7 | 3.21 | 41 | 95.4 | 82.7 | 66.6 | 48.2 | 30.1 | 11.0 |
| C3-10-MFT-L | 302 | 43.5 | 3.69 | 49 | 95.4 | 82.1 | 65.4 | 47.7 | 31.2 | 12.5 |
| | | | | | | | | | | |
| C3-11-CapW | 334 | | | | | | | | | |
| C3-11-MFT-U | 334 | 45.7 | 3.69 | 67.6 | 85.5 | 73.4 | 58.1 | 41.3 | 25.6 | 9.1 |
| C3-11-MFT-L | 334 | 46.2 | 3.93 | 69.8 | 89.8 | 77.5 | 61.7 | 44.2 | 27.6 | 9.8 |
| | | | | | | | | | | |
| C3-12-CapW | 365 | | | | | | | | | |
| C3-12-MFT-U | 365 | 44.3 | 3.67 | 69.7 | 92.0 | 79.6 | 63.8 | 46.0 | 28.8 | 10.4 |
| C3-12-MFT-L | 365 | 45.6 | 3.67 | 77.1 | 92.2 | 79.9 | 64.2 | 46.5 | 29.3 | 10.7 |

1). NA=not available.
2). BDL=below detection limits.

**Table B-8. Physical and chemical analyses in System 4.**

| | | WATER PROPERTIES | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | |
| Sample | $t_{incu}$ (d) | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C4-1-CapW | 28 | 7.9 | 6320 | 270 | NA | BDL | 1330 | 19.2 | 27.5 | 60 | BDL | 1100 | 1070 | 642 | BDL | 2.9 | BDL | BDL | 3.9 | 1.57 |
| C4-1-CT-U | 28 | 8.2 | 6040 | 280 | 5.9 | BDL | 1200 | 14.7 | 17.5 | 34.9 | BDL | 1100 | 807 | 950 | BDL | 2.3 | BDL | BDL | 2.9 | 0.85 |
| C4-1-CT-L | 28 | 8.2 | 5820 | 270 | 8.5 | BDL | 1260 | 15.3 | 16.9 | 34.1 | BDL | 1100 | 659 | 1010 | BDL | 2.64 | 0.10 | BDL | 3.7 | 0.85 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-2-CapW | 61 | 8.1 | 6230 | 270 | 7.8 | 3 | 1350 | 19.2 | 26.6 | 34.7 | 15.0 | 1100 | 814 | 1100 | BDL | 1.6 | BDL | BDL | 4.1 | 1.36 |
| C4-2 CT-U | 61 | 8 | 5520 | 260 | 6.1 | 0.1 | 1190 | 14.5 | 13.3 | 26.4 | BDL | 1000 | 497 | 1220 | BDL | 2.91 | 0.37 | BDL | 4.2 | 0.76 |
| C4-2 CT-L | 61 | 8 | 5340 | 260 | 5.2 | 0.1 | 1200 | 14.2 | 12.6 | 25.0 | BDL | 1000 | 413 | 1230 | BDL | 2.78 | 0.57 | BDL | 4.2 | 0.74 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-3-CapW | 90 | 8 | 6360 | 250 | 7.3 | 1 | 1370 | 17.7 | 25.7 | 39 | BDL | 1000 | 1040 | 1000 | BDL | 2.82 | BDL | BDL | 4.1 | 1.37 |
| C4-3 CT-U | 90 | 8.1 | 5450 | 260 | 4.1 | 0.1 | 1170 | 13.7 | 12.5 | 23.5 | BDL | 1000 | 355 | 1140 | BDL | 2.65 | 0.34 | BDL | 3.6 | 0.70 |
| C4-3 CT-L | 90 | 8.1 | 5410 | 260 | 5.1 | 0.3 | 1170 | 13.7 | 11.9 | 22.6 | BDL | 1000 | 381 | 1090 | BDL | 2.73 | 0.47 | BDL | 3.8 | 0.70 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-4-CapW | 120 | 7.78 | 6300 | 260 | 6.7 | BDL | 1320 | 17.4 | 25.4 | 50.3 | BDL | 1000 | 927 | 1080 | BDL | 2.94 | BDL | BDL | 4.1 | 1.36 |
| C4-4-CT-U | 120 | 8.1 | 5410 | 260 | 5.0 | 0.1 | 1190 | 13.2 | 13.8 | 27.4 | BDL | 1000 | 164 | 1220 | BDL | 2.42 | 0.34 | BDL | 3.6 | 0.79 |
| C4-4-CT-L | 120 | 8.1 | 5390 | 260 | 5.0 | 0.1 | 1170 | 13.1 | 13.5 | 27.0 | BDL | 1000 | 75 | 1280 | BDL | 2.61 | 0.48 | BDL | 3.9 | 0.80 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-5-CapW | 152 | NA | NA | NA | NA | 0.8 | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA |
| C4-5-CT-U | 152 | 8.01 | 5250 | 260 | 5.7 | 0.4 | 1090 | 12.5 | 12.3 | 23.7 | BDL | 1000 | 404 | 1144 | BDL | 2.51 | 0.29 | BDL | 3.67 | 0.70 |
| C4-5-CT-L | 152 | 8.05 | 5180 | 250 | 5.5 | 0.2 | 1060 | 12.3 | 12.2 | 23.4 | BDL | 990 | 339 | 1134 | BDL | 2.60 | 0.48 | BDL | 3.94 | 0.72 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-6-CapW | 181 | 7.76 | 6190 | 260 | 6.65 | BDL | 1370 | 18.6 | 25.9 | 55.6 | BDL | 1000 | 873 | 1124 | BDL | 2.86 | BDL | BDL | 4.13 | 1.46 |
| C4-6-CT-U | 181 | 8.05 | 5420 | 250 | NA | 0.8 | 1210 | 15.5 | 13.6 | 26.5 | BDL | 1000 | 348 | 1044 | BDL | 2.81 | 0.35 | BDL | 4.01 | 0.81 |
| C4-6-CT-L | 181 | 8.08 | 5240 | 250 | NA | 0.6 | 1170 | 14.5 | 12.6 | 23.6 | BDL | 970 | 61.4 | 1064 | BDL | 3.06 | 0.54 | BDL | 4.42 | 0.77 |

**Table B-8.** *(continued).*

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C4-7-CapW | 212 | 7.9 | 6240 | 250 | 8.7 | 0.2 | 1530 | 18.8 | 24.5 | 31.5 | BDL | 1000 | 702 | 1340 | BDL | 3.28 | 0.07 | BDL | 5.00 | 1.26 |
| C4-7-CT-U | 212 | 8 | 5320 | 260 | 7.6 | 0.2 | 1350 | 13.7 | 12.6 | 22.8 | BDL | 1000 | 301 | 1200 | BDL | 3.08 | 0.57 | BDL | 4.47 | 0.73 |
| C4-7-CT-L | 212 | 8.1 | 5440 | 270 | 6.9 | 0.1 | 1400 | 13.7 | 13.7 | 23.2 | BDL | 1000 | 310 | 1230 | BDL | 2.57 | 0.27 | BDL | 3.75 | 0.74 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-8-CapW | 243 | 8 | 6100 | 260 | 8.4 | 1 | 1450 | 18.2 | 21.9 | 19.7 | BDL | 1000 | 490 | 1480 | BDL | 3.07 | 0.02 | BDL | 4.18 | 1.09 |
| C4-8-CT-U | 243 | 8.1 | 5370 | 260 | 7.1 | BDL | 1290 | 13.6 | 12.9 | 23.3 | BDL | 1000 | 256 | 1140 | BDL | 2.71 | 0.56 | BDL | 3.93 | 0.77 |
| C4-8-CT-L | 243 | 7.9 | 5450 | 260 | 6.9 | BDL | 1340 | 14.5 | 12.7 | 23.1 | BDL | 1000 | 260 | 1210 | BDL | 3.09 | 0.41 | BDL | 4.36 | 0.76 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-9-CapW | 266 | 7.73 | 6340 | 270 | 8.0 | BDL | 1420 | 16.6 | 22.4 | 35.8 | BDL | 1000 | 764 | 1360 | BDL | 2.81 | BDL | BDL | 3.88 | 1.18 |
| C4-9-CT-U | 266 | 7.90 | 5620 | 270 | 7.1 | BDL | 1270 | 12.7 | 11.9 | 20.8 | BDL | 1000 | 359 | 1220 | BDL | 2.66 | 0.21 | BDL | 3.44 | 0.66 |
| C4-9-CT-L | 266 | 8.01 | 5520 | 270 | 7.0 | BDL | 1280 | 12.5 | 11.0 | 17.9 | BDL | 1000 | 305 | 1260 | BDL | 2.76 | 0.37 | BDL | 4.05 | 0.63 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-10-CapW | 302 | 7.6 | 6240 | 270 | 7.7 | 0.4 | 1550 | 18.3 | 25.1 | 44.4 | BDL | 1000 | 712 | 1300 | BDL | 3.16 | 0.05 | BDL | 4.52 | 1.36 |
| C4-10-CT-U | 302 | 8 | 5540 | 270 | 7.1 | BDL | 1320 | 13.3 | 13.3 | 24.0 | BDL | 1000 | 226 | 1230 | BDL | 2.41 | 0.33 | BDL | 3.77 | 0.75 |
| C4-10-CT-L | 302 | 8 | 5340 | 270 | NA | BDL | 1300 | 12.8 | 12.5 | 22.7 | BDL | 1000 | 192 | 1230 | BDL | 2.56 | 0.44 | BDL | 3.92 | 0.72 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-11-CapW | 334 | 7.9 | 6300 | 260 | NA | BDL | 1420 | 20.4 | 29.5 | 67.7 | BDL | 1000 | 965 | 1120 | BDL | 3.58 | 0.14 | BDL | 5.22 | 1.66 |
| C4-11-CT-U | 334 | 8.1 | 5550 | 260 | NA | 1 | 1170 | 14.0 | 14.2 | 27.0 | BDL | 1000 | 316 | 1200 | BDL | 2.89 | 0.27 | BDL | 4.24 | 0.77 |
| C4-11-CT-L | 334 | 8.2 | 5290 | 260 | NA | BDL | 1110 | 13.0 | 12.7 | 23.0 | BDL | 1000 | 172 | 1270 | BDL | 2.61 | 0.46 | BDL | 4.28 | 0.70 |
| | | | | | | | | | | | | | | | | | | | | |
| C4-12-CapW | 365 | 7.7 | 6440 | 260 | 7.6 | BDL | 1570 | 19.7 | 26.7 | 57.5 | BDL | 1000 | 833 | 1260 | BDL | 3.48 | 0.03 | BDL | 5.07 | 1.53 |
| C4-12-CT-U | 365 | 8.1 | 5580 | 260 | 5.9 | 4 | 1370 | 15.2 | 13.9 | 25.4 | BDL | 1000 | 259 | 1290 | BDL | 3.15 | 0.27 | BDL | 4.87 | 0.79 |
| C4-12-CT-L | 365 | 8.1 | 5370 | 260 | 6.9 | 1 | 1410 | 15.1 | 13.5 | 24.7 | BDL | 1000 | 178 | 1350 | BDL | 3.34 | 0.45 | BDL | 5.34 | 0.78 |

**Table B-8.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | Particle Size Distribution (% of solids less than) | | | | | |
| | | | | | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| C4-1-CapW | 28 | | | | | | | | | |
| C4-1-CT-U | 28 | 65.0 | 1.31 | -197 | 47.0 | 41.0 | 32.5 | 19.7 | 10.5 | 3.7 |
| C4-1-CT-L | 28 | 69.9 | 1.50 | -199 | 39.2 | 31.8 | 24.2 | 17.2 | 11.5 | 5.26 |
| | | | | | | | | | | |
| C4-2-CapW | 61 | | | | | | | | | |
| C4-2 CT-U | 61 | 69.3 | 1.35 | -217 | 41.3 | 34.3 | 27.0 | 19.6 | 12.9 | 5.30 |
| C4-2 CT-L | 61 | 70.9 | 1.36 | -210 | 37.9 | 31.5 | 24.9 | 18.1 | 11.8 | 4.68 |
| | | | | | | | | | | |
| C4-3-CapW | 90 | | | | | | | | | |
| C4-3 CT-U | 90 | 70.7 | 1.36 | -205 | 37.3 | 31.3 | 24.9 | 18.2 | 11.9 | 4.83 |
| C4-3 CT-L | 90 | 72.6 | 1.32 | -208 | 36.6 | 30.9 | 24.7 | 18.1 | 11.5 | 4.29 |
| | | | | | | | | | | |
| C4-4-CapW | 120 | | | | | | | | | |
| C4-4-CT-U | 120 | 71.3 | 1.15 | -209 | 38.2 | 31.8 | 25.1 | 18.3 | 11.9 | 4.72 |
| C4-4-CT-L | 120 | 73.3 | 1.36 | -207 | 35.4 | 29.4 | 23.3 | 16.9 | 11.0 | 4.42 |
| | | | | | | | | | | |
| C4-5-CapW | 152 | | | | | | | | | |
| C4-5-CT-U | 152 | 71.2 | 1.42 | -206 | 48.2 | 40.1 | 31.6 | 22.9 | 14.7 | 5.16 |
| C4-5-CT-L | 152 | 73.6 | 1.33 | -201 | 41.3 | 34.5 | 27.2 | 19.8 | 12.7 | 4.57 |
| | | | | | | | | | | |
| C4-6-CapW | 181 | | | | | | | | | |
| C4-6-CT-U | 181 | 70.4 | 1.41 | -196 | 37.4 | 31.2 | 24.4 | 17.6 | 11.3 | 4.24 |
| C4-6-CT-L | 181 | 72.8 | 1.45 | -201 | 39.9 | 33.3 | 26.2 | 18.9 | 12.2 | 4.48 |

**Table B-8.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | Particle Size Distribution (% of solids less than) | | | | | |
| | | | | | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| C4-7-CapW | 212 | | | | | | | | | |
| C4-7-CT-U | 212 | 75.7 | 1.44 | -205 | 31 | 26.5 | 21.2 | 15.5 | 10.3 | 4.35 |
| C4-7-CT-L | 212 | 74.1 | 1.50 | -194 | 32 | 26.2 | 20.6 | 14.7 | 9.6 | 4.06 |
| | | | | | | | | | | |
| C4-8-CapW | 243 | | | | | | | | | |
| C4-8-CT-U | 243 | 74.0 | 1.43 | -204.6 | 38 | 30.8 | 24.0 | 16.8 | 10.4 | 4.08 |
| C4-8-CT-L | 243 | 73.0 | 1.44 | -195.3 | 38 | 32.0 | 25.3 | 18.5 | 12.4 | 5.41 |
| | | | | | | | | | | |
| C4-9-CapW | 266 | | | | | | | | | |
| C4-9-CT-U | 266 | 71.5 | 1.24 | -201.1 | 33 | 27.6 | 22.3 | 16.0 | 9.8 | 2.80 |
| C4-9-CT-L | 266 | 73.2 | 1.24 | -204.9 | 33 | 27.5 | 21.8 | 15.8 | 10.1 | 3.06 |
| | | | | | | | | | | |
| C4-10-CapW | 302 | | | | | | | | | |
| C4-10-CT-U | 302 | 71.9 | 1.18 | -190 | 36.7 | 30.9 | 24.8 | 17.9 | 11.0 | 4.1 |
| C4-10-CT-L | 302 | 73.5 | 1.35 | -200 | 30.1 | 25.5 | 20.6 | 15.4 | 10.2 | 4.0 |
| | | | | | | | | | | |
| C4-11-CapW | 334 | | | | | | | | | |
| C4-11-CT-U | 334 | 74.9 | 1.52 | -184.5 | 33.0 | 27.1 | 21.1 | 15.0 | 9.6 | 3.73 |
| C4-11-CT-L | 334 | 75.9 | 1.49 | -196.7 | 32.9 | 27.2 | 21.2 | 15.2 | 9.9 | 3.89 |
| | | | | | | | | | | |
| C4-12-CapW | 365 | | | | | | | | | |
| C4-12-CT-U | 365 | 75.9 | 0.83 | -168 | 38.4 | 33.1 | 27.1 | 18.7 | 10.4 | 3.73 |
| C4-12-CT-L | 365 | 76.4 | 1.14 | -188 | 39.6 | 33.6 | 27.2 | 19.7 | 11.9 | 4.08 |

1). NA=not available; 2). BDL=below detection limits.

**Table B-9. Physical and chemical analyses in System 1.**

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C1-2-CapW | 3 | 8.1 | 4610 | 25 | 10.2 | BDL | 1030 | 16 | 16.9 | 24 | BDL | 880 | 115 | 1449 | BDL | 3.8 | 0.44 | BDL | 5 | 1.01 |
| C1-2-MFT-U | 3 | 8 | 4490 | 23 | NA | BDL | 977 | 14 | 14 | 20 | BDL | 870 | 125 | | BDL | 2.7 | 0.34 | BDL | 3.7 | 0.80 |
| C1-2-MFT-L | 3 | 7.9 | 4730 | 77 | NA | BDL | 1000 | 15 | 14.7 | 21 | BDL | 920 | 226 | 1163 | BDL | 2.4 | 0.27 | BDL | 3.2 | 0.84 |
| C1-2-CT-U | 3 | 8.1 | 5610 | 210 | NA | BDL | 1130 | 17 | 19.9 | 51 | BDL | 1000 | 682 | 752 | BDL | 2.3 | 0.11 | BDL | 3 | 1.12 |
| C1-2-CT-L | 3 | 8.2 | 5400 | 210 | NA | BDL | 1140 | 17 | 19.9 | 51 | BDL | 1000 | 690 | 690 | BDL | 2.3 | 0.11 | BDL | 3 | 1.11 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-3-CapW | 15 | 7.8 | 4850 | 66 | 8.2 | BDL | 1060 | 16 | 17.5 | 27 | BDL | 930 | 235 | 1338 | BDL | 3.5 | 0.43 | BDL | 4.9 | 1.10 |
| C1-3-MFT-U | 15 | 8 | 4460 | 50 | NA | BDL | 964 | 15 | 12.1 | 17 | BDL | 890 | 51.2 | 1342 | BDL | 2.9 | 0.36 | BDL | 3.9 | 0.71 |
| C1-3-MFT-L | 15 | 8 | 4780 | 120 | NA | BDL | 1010 | 15 | 13.7 | 19 | BDL | 950 | 136 | 1235 | BDL | 2.5 | 0.34 | BDL | 3.7 | 0.80 |
| C1-3-CT-U | 15 | 8.1 | 5380 | 240 | NA | BDL | 1120 | 15 | 15.9 | 36 | BDL | 1000 | 491 | 818 | BDL | 2.4 | 0.09 | BDL | 3.3 | 0.85 |
| C1-3-CT-L | 15 | 8.2 | 5380 | 240 | NA | BDL | 1110 | 15 | 17.9 | 44 | BDL | 1000 | 503 | 812 | BDL | 2.5 | 0.13 | BDL | 3.7 | 0.97 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-4-CapW | 29 | 8 | 4820 | 86 | NA | BDL | 1040 | 16 | 17.4 | 28 | BDL | 910 | 229 | 1301 | BDL | 3.4 | 0.41 | BDL | BDL | 1.07 |
| C1-4-MFT-U | 29 | 8 | 4570 | 82 | 8.1 | BDL | 997 | 15.2 | 12.9 | 17.5 | BDL | 940 | 31 | 1336 | BDL | 1.1 | 0.39 | BDL | 3.98 | 0.77 |
| C1-4-MFT-L | 29 | 8.1 | 4840 | 160 | 8.6 | BDL | 1050 | 15.3 | 13.7 | 18.8 | BDL | 1000 | 87 | 1232 | BDL | 0.8 | 0.40 | BDL | 3.73 | 0.81 |
| C1-4-CT-U | 29 | 8.4 | 5090 | 270 | 4.7 | BDL | 1070 | 14.6 | 14.2 | 29.0 | 6.50 | 1100 | 336 | 938 | BDL | 0.7 | 0.67 | BDL | 3.48 | 0.73 |
| C1-4-CT-L | 29 | 8.4 | 5130 | 270 | 4.1 | BDL | 1060 | 15.0 | 15.0 | 23.2 | BDL | 1000 | 260 | 878 | BDL | 0.9 | 0.53 | BDL | 3.69 | 0.73 |

Table B-9. *(continued).*

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | pH | EC (uS/cm) | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | | | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C1-5-CapW | 62 | 7.6 | 4750 | 70 | 9.0 | 0.0 | 1060 | 16.2 | 17.2 | 25.6 | BDL | 900 | 73 | NA | BDL | 2.4 | 0.52 | BDL | 5.29 | 1.08 |
| C1-5-MFT-U | 62 | 8.1 | 4660 | 130 | 8.2 | 0.0 | 1010 | 14.2 | 12.3 | 16.5 | BDL | 920 | 29.6 | 1380 | BDL | 2.65 | 0.36 | BDL | 4.03 | 0.74 |
| C1-5-MFT-L | 62 | 8.3 | 4990 | 210 | 8.2 | 0.0 | 1040 | 13.7 | 13.4 | 17.8 | BDL | 990 | 106.0 | 1279 | BDL | 2.28 | 0.34 | BDL | 3.58 | 0.76 |
| C1-5-CT-U | 62 | 8.3 | 5120 | 260 | 5.5 | 0.0 | 1070 | 13.8 | 13.5 | 28.6 | BDL | 1000 | 286.0 | 960 | BDL | 2.56 | 0.26 | BDL | 3.79 | 0.75 |
| C1-5-CT-L | 62 | 8 | 5140 | 260 | 4.3 | 0.1 | 1090 | 13.3 | 12.9 | 25.8 | BDL | 1000 | 274.0 | 955 | BDL | 2.70 | 0.42 | BDL | 4.02 | 0.74 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-6-CapW | 91 | 7.7 | 4560 | 77 | NA | 0.0 | 974 | 14.6 | 16.3 | 24.1 | BDL | 840 | BDL | 1538 | BDL | 3.49 | 0.49 | BDL | 5.29 | 1.01 |
| C1-6-MFT-U | 91 | 8.1 | 4730 | 120 | 8.7 | 0.0 | 1030 | 14.5 | 12.7 | 16.8 | BDL | 920 | 17.3 | 1397 | BDL | 2.42 | 0.38 | BDL | 3.34 | 0.75 |
| C1-6-MFT-L | 91 | 8.2 | 4860 | 200 | 8.4 | 0.0 | 1060 | 13.9 | 12.9 | 16.6 | BDL | 970 | 63.1 | 1260 | BDL | 2.18 | 0.34 | BDL | 3.15 | 0.73 |
| C1-6-CT-U | 91 | 8.1 | 5000 | 260 | 5.7 | 0.0 | 1050 | 12.8 | 12.1 | 22.8 | BDL | 1000 | 177.0 | 1040 | BDL | 2.41 | 0.41 | BDL | 3.42 | 0.68 |
| C1-6-CT-L | 91 | 8.2 | 5090 | 280 | 4.3 | 0.2 | 1080 | 13.6 | 12.7 | 26.3 | BDL | 1000 | 201.0 | 1020 | BDL | 2.60 | 0.42 | BDL | 3.62 | 0.74 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-12-CapW | 121 | 7.71 | 4870 | 92 | 9.0 | 0.0 | 1050 | 14.9 | 16.8 | 24.2 | BDL | 910 | 90.3 | 1550 | BDL | 3.42 | 0.37 | BDL | 5.03 | 1.02 |
| C1-12-MFT-U | 121 | 7.9 | 4790 | 140 | 8.5 | 0.0 | 1080 | 14.0 | 13.3 | 16.9 | BDL | 940 | 40.7 | 1405 | BDL | 2.29 | 0.34 | BDL | 3.74 | 0.77 |
| C1-12-MFT-L | 121 | 8.1 | 4980 | 190 | 7.6 | 0.0 | 1150 | 14.2 | 15.0 | 20.3 | BDL | 990 | 94.1 | 1340 | BDL | 2.28 | 0.35 | BDL | 3.75 | 0.85 |
| C1-12-CT-U | 121 | 8 | 5230 | 250 | 4.2 | 0.1 | 1140 | 13.6 | 15.1 | 33.0 | BDL | 1000 | 264.0 | 1120 | BDL | 2.66 | 0.38 | BDL | 4.06 | 0.87 |
| C1-12-CT-L | 121 | 8 | 5110 | 240 | 5.0 | 0.1 | 1110 | 12.7 | 13.8 | 28.3 | BDL | 1000 | 67.5 | 1151 | BDL | 2.54 | 0.44 | BDL | 3.87 | 0.80 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-8-CapW | 153 | NA | NA | NA | NA | BDL | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA | NA |
| C1-8-MFT-U | 153 | 7.85 | 4280 | 130 | 15.6 | 0.1 | 918 | 13.1 | 11.5 | 14.8 | BDL | 900 | 12 | 1382 | BDL | 2.36 | 0.32 | BDL | 3.87 | 0.68 |
| C1-8-MFT-L | 153 | 7.95 | 4500 | 200 | 8.4 | 3.0 | 956 | 13.0 | 12.5 | 17.2 | BDL | 970 | 59 | 1328 | BDL | 2.31 | 0.33 | BDL | 3.79 | 0.73 |
| C1-8-CT-U | 153 | 7.85 | 4390 | 250 | 5.9 | 1.0 | 945 | 11.9 | 11.6 | 23.4 | BDL | 1000 | 151 | 1132 | BDL | 2.57 | 0.43 | BDL | 3.92 | 0.68 |
| C1-8-CT-L | 153 | 8.03 | 4650 | 280 | 5.3 | 0.8 | 950 | 12.1 | 11.5 | 23.8 | BDL | 1000 | 159 | 1076 | BDL | 2.47 | 0.45 | BDL | 3.66 | 0.69 |

**Table B-9.** *(continued).*

| Sample | $t_{inco}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C1-9-CapW | 182 | 7.68 | 4890 | 97 | 9.2 | 1.5 | 1090 | 15.9 | 17.3 | 26.0 | BDL | 880 | 68.3 | 1596 | BDL | 3.51 | 0.45 | BDL | 5.11 | 1.08 |
| C1-9-MFT-U | 182 | 7.98 | 4720 | 127 | NA | 0.6 | 1050 | 14.1 | 12.9 | 16.9 | BDL | 900 | 22.6 | 1421 | BDL | 2.59 | 0.36 | BDL | 4.27 | 0.77 |
| C1-9-MFT-L | 182 | 8.10 | 4900 | 188 | NA | 3.5 | 1110 | 14.8 | 14.3 | 20.2 | BDL | 960 | 54.6 | 1382 | BDL | 2.54 | 0.41 | BDL | 4.31 | 0.85 |
| C1-9-CT-U | 182 | 8.00 | 4920 | 245 | NA | 6.0 | 1130 | 14.6 | 13.1 | 27.1 | BDL | 990 | 70.0 | 1170 | BDL | 2.97 | 0.54 | BDL | 4.63 | 0.80 |
| C1-9-CT-L | 182 | 8.15 | 4920 | 270 | NA | 5.5 | 1110 | 15.4 | 13.5 | 26.0 | BDL | 1000 | 35.1 | 1124 | BDL | 2.88 | 0.74 | BDL | 4.18 | 0.81 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-10-CapW | 213 | 7.7 | 4870 | 110 | 11.7 | BDL | 1230 | 16.6 | 17.2 | 25.1 | BDL | 900 | 52.6 | 1580 | BDL | 3.92 | 0.50 | BDL | 5.59 | 1.11 |
| C1-10-MFT-U | 213 | 8.2 | 4550 | 150 | 9.2 | BDL | 1130 | 13.5 | 10.6 | 11.8 | BDL | 950 | 18.8 | 1192 | BDL | 2.32 | 0.30 | BDL | 3.30 | 0.54 |
| C1-10-MFT-L | 213 | 8.2 | 4860 | 210 | 9.7 | 3 | 1230 | 14.1 | 12.9 | 15.9 | 4.2 | 1000 | 61.3 | 1270 | BDL | 2.70 | 0.35 | BDL | 3.87 | 0.69 |
| C1-10-CT-U | 213 | 7.9 | 5270 | 260 | 7.1 | 0.2 | 1330 | 14.0 | 14.3 | 26.0 | BDL | 1000 | 226 | 1210 | BDL | 3.11 | 0.58 | BDL | 4.37 | 0.81 |
| C1-10-CT-L | 213 | 8 | 5260 | 270 | 3.3 | 0.1 | 1320 | 14.3 | 14.8 | 28.6 | BDL | 1000 | 267 | 1140 | BDL | 3.09 | 0.47 | BDL | 4.48 | 0.83 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-11-CapW | 244 | 7.6 | 4960 | 110 | 10.3 | BDL | 1200 | 16.4 | 17.2 | 25.1 | BDL | 860 | 133 | 1460 | BDL | 3.68 | 0.53 | BDL | 5.34 | 1.10 |
| C1-11-MFT-U | 244 | 8 | 4650 | 140 | 8.1 | 2 | 1150 | 13.5 | 11.6 | 13.4 | BDL | 910 | 33 | 1230 | BDL | 2.64 | 0.32 | BDL | 4.07 | 0.67 |
| C1-11-MFT-L | 244 | 8.1 | 4850 | 190 | 7.9 | 5 | 1170 | 13.9 | 12.6 | 15.2 | BDL | 950 | 58 | 1220 | BDL | 2.59 | 0.35 | BDL | 4.01 | 0.72 |
| C1-11-CT-U | 244 | 7.9 | 5140 | 220 | 6.5 | BDL | 1210 | 13.7 | 13.6 | 25.6 | BDL | 900 | 110 | 1159 | BDL | 2.70 | 0.55 | BDL | 4.01 | 0.80 |
| C1-11-CT-L | 244 | 8.1 | 5230 | 270 | 4.8 | BDL | 1270 | 14.5 | 15.4 | 30.3 | BDL | 1000 | 95 | 1250 | BDL | 2.87 | 0.53 | BDL | 4.28 | 0.91 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-13-CapW | 267 | 7.54 | 5000 | 110 | 10.5 | BDL | 1150 | 15.1 | 16.1 | 22.9 | BDL | 900 | 188 | 1440 | BDL | 3.40 | 0.38 | BDL | 4.92 | 1.02 |
| C1-13-MFT-U | 267 | 7.93 | 4660 | 150 | 8.8 | 3 | 1110 | 12.4 | 10.8 | 12.3 | BDL | 940 | 45 | 1240 | BDL | 2.28 | 0.23 | BDL | 3.27 | 0.59 |
| C1-13-MFT-L | 267 | 7.95 | 4780 | 190 | 8.0 | 4 | 1130 | 12.1 | 11.3 | 14.5 | BDL | 970 | 66 | 1260 | BDL | 2.37 | 0.26 | BDL | 3.38 | 0.62 |
| C1-13-CT-U | 267 | 7.76 | 4970 | 230 | 7.1 | BDL | 1180 | 12.8 | 11.9 | 21.4 | BDL | 1000 | 102 | 1270 | BDL | 2.87 | 0.37 | BDL | 4.31 | 0.69 |
| C1-13-CT-L | 267 | 7.91 | 5100 | 250 | 7.1 | 0.4 | 1180 | 13.6 | 12.6 | 24.2 | BDL | 1000 | 90 | 1260 | BDL | 3.02 | 0.41 | BDL | 4.46 | 0.75 |

**Table B-9.** *(continued).*

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C1-14-CapW | 303 | 7.4 | 5270 | 120 | 10.4 | BDL | 1230 | 16.3 | 19.1 | 30.2 | BDL | 910 | 277 | NA | BDL | 3.54 | 0.52 | BDL | 5.37 | 1.20 |
| C1-14-MFT-U | 303 | 7.8 | 4910 | 150 | 9.1 | BDL | 1170 | 13.6 | 12.9 | 16.1 | BDL | 940 | 45.0 | NA | BDL | 2.66 | 0.36 | BDL | 4.39 | 0.73 |
| C1-14-MFT-L | 303 | 7.9 | 4980 | 180 | NA | 3 | 1190 | 13.3 | 12.9 | 16.6 | BDL | 970 | 60.3 | NA | BDL | 2.68 | 0.35 | BDL | 4.04 | 0.73 |
| C1-14-CT-U | 303 | 8 | 5320 | 240 | 7.9 | BDL | 1360 | 14.5 | 14.9 | 28.4 | BDL | 1000 | 156 | NA | BDL | 2.87 | 0.46 | BDL | 4.53 | 0.85 |
| C1-14-CT-L | 303 | 8.1 | 5330 | 260 | NA | BDL | 1360 | 14.6 | 16.1 | 30.9 | BDL | 1000 | 80.3 | NA | BDL | 2.96 | 0.60 | BDL | 4.53 | 0.95 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-15-CapW | 335 | 7.7 | 5100 | 110 | NA | BDL | 1080 | 16.6 | 19.4 | 29.3 | BDL | 920 | 237 | 1430 | BDL | 3.80 | 0.50 | BDL | 5.72 | 1.17 |
| C1-15-MFT-U | 335 | 8.1 | 4750 | 140 | NA | 2 | 1090 | 13.7 | 12.5 | 14.4 | BDL | 960 | 22 | 1300 | BDL | 2.70 | 0.34 | BDL | 4.46 | 0.67 |
| C1-15-MFT-L | 335 | 8.1 | 4810 | 160 | NA | 4 | 1040 | 13.2 | 12.5 | 15.9 | BDL | 970 | 41 | 1310 | BDL | 2.72 | 0.33 | BDL | 4.17 | 0.67 |
| C1-15-CT-U | 335 | 8.3 | 5220 | 220 | NA | BDL | 1090 | 14.0 | 13.3 | 24.1 | BDL | 1000 | 53 | 1350 | BDL | 3.26 | 0.48 | BDL | 5.14 | 0.74 |
| C1-15-CT-L | 335 | 8.2 | 5330 | 240 | NA | BDL | 1120 | 14.5 | 14.4 | 28.7 | BDL | 1000 | 108 | 1330 | BDL | 3.21 | 0.54 | BDL | 5.18 | 0.83 |
| | | | | | | | | | | | | | | | | | | | | |
| C1-16-CapW | 366 | 8.1 | 5150 | 110 | 9.6 | BDL | 1280 | 17.0 | 17.9 | 26.8 | BDL | 910 | 271 | 1380 | BDL | 3.78 | 0.00 | BDL | 5.16 | 1.13 |
| C1-16-MFT-U | 366 | 8 | 4830 | 150 | 7.8 | 3 | 1170 | 13.0 | 11.8 | 13.0 | BDL | 950 | 44 | 1320 | BDL | 2.67 | 0.26 | BDL | 3.82 | 0.64 |
| C1-16-MFT-L | 366 | 8 | 4940 | 180 | 7.9 | 3.5 | 1150 | 12.8 | 12.1 | 14.3 | BDL | 990 | 60 | 1320 | BDL | 2.64 | 0.27 | BDL | 3.88 | 0.65 |
| C1-16-CT-U | 366 | 8.2 | 5350 | 240 | 7.5 | BDL | 1260 | 14.1 | 14.9 | 20.9 | BDL | 1000 | 21 | 1350 | BDL | 3.35 | 0.42 | BDL | 5.19 | 0.80 |
| C1-16-CT-L | 366 | 8 | 5190 | 230 | 7.5 | BDL | 1240 | 13.9 | 12.5 | 18.1 | BDL | 1000 | 23 | 1310 | BDL | 3.20 | 0.38 | BDL | 4.73 | 0.69 |

**Table B-9.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | Particle Size Distribution (% of solids less than) | | | | | |
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | AVS (mg/kg of dry sample) | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| C1-2-CapW | 3 | | | 401 | | | | | | | |
| C1-2-MFT-U | 3 | 38.8 | 3.05 | 42 | | 91.4 | 78.4 | 61.9 | 44.6 | 28.60 | 10.60 |
| C1-2-MFT-L | 3 | 40.9 | 3.36 | 33 | | 90.5 | 77.3 | 60.1 | 42.8 | 27.90 | 11.30 |
| C1-2-CT-U | 3 | 60.8 | 1.09 | 204 | | 34.2 | 28.0 | 21.8 | 15.8 | 10.30 | 3.94 |
| C1-2-CT-L | 3 | 63.2 | 1.10 | -41 | | 37.4 | 30.8 | 24.0 | 17.3 | 11.10 | 4.26 |
| | | | | | | | | | | | |
| C1-3-CapW | 15 | | | 558 | | | | | | | |
| C1-3-MFT-U | 15 | 39.5 | 3.23 | 4 | | 90.3 | 77.2 | 60.6 | 43.4 | 27.90 | 10.80 |
| C1-3-MFT-L | 15 | 39.2 | 3.26 | -50 | | 92.7 | 79.3 | 62.3 | 44.6 | 28.70 | 11.10 |
| C1-3-CT-U | 15 | 63.3 | 1.23 | -184 | | 29.0 | 24.0 | 18.7 | 13.4 | 8.75 | 3.50 |
| C1-3-CT-L | 15 | 72.4 | 1.38 | -195 | | 40.3 | 33.2 | 25.7 | 18.3 | 12.10 | 5.01 |
| | | | | | | | | | | | |
| C1-4-CapW | 29 | | | | | | | | | | |
| C1-4-MFT-U | 29 | 38.0 | 2.99 | -116 | | 90.7 | 77.9 | 61.2 | 43.9 | 28.2 | 10.9 |
| C1-4-MFT-L | 29 | 38.3 | 3.25 | -114 | | 89.8 | 76.8 | 60.4 | 43.1 | 27.3 | 10.2 |
| C1-4-CT-U | 29 | 66.2 | 1.00 | -191 | | 40.6 | 34.1 | 26.9 | 19.4 | 12.5 | 4.7 |
| C1-4-CT-L | 29 | 72.3 | 1.30 | -190 | | 33.4 | 27.8 | 21.6 | 15.5 | 10.1 | 4.1 |

**Table B-9.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | AVS (mg/kg of dry sample) | Particle Size Distribution (% of solids less than) | | | | | |
| | | | | | | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| C1-5-CapW | 62 | | | | | | | | | | |
| C1-5-MFT-U | 62 | 38.3 | 3.26 | -110 | | 99.2 | 84.5 | 66.5 | 48.1 | 31.6 | 12.70 |
| C1-5-MFT-L | 62 | 38.8 | 3.04 | -180 | | 95.4 | 82.6 | 66.3 | 48.6 | 31.3 | 11.40 |
| C1-5-CT-U | 62 | 71.4 | 1.36 | -194 | | 42.6 | 35.5 | 28.0 | 20.3 | 13.2 | 5.29 |
| C1-5-CT-L | 62 | 69.2 | 1.28 | -199 | | 42.9 | 35.7 | 28.2 | 20.4 | 13.2 | 5.12 |
| | | | | | | | | | | | |
| C1-6-CapW | 91 | | | | | | | | | | |
| C1-6-MFT-U | 91 | 38.9 | 3.19 | -164 | | 95.7 | 82.6 | 65.9 | 47.9 | 31.1 | 12.10 |
| C1-6-MFT-L | 91 | 39.1 | 3.33 | -178 | | 95.2 | 81.7 | 64.7 | 46.8 | 30.8 | 12.50 |
| C1-6-CT-U | 91 | 70.7 | 1.33 | -197 | | 39.0 | 32.3 | 25.4 | 18.5 | 12.1 | 4.90 |
| C1-6-CT-L | 91 | 72.0 | 1.36 | -197 | | 28.3 | 23.3 | 18.3 | 13.4 | 8.9 | 3.69 |
| | | | | | | | | | | | |
| C1-12-CapW | 121 | | | | | | | | | | |
| C1-12-MFT-U | 121 | 38.9 | 3.18 | -135 | 150.4 | 92.5 | 80.3 | 64.3 | 46.4 | 29.1 | 10.40 |
| C1-12-MFT-L | 121 | 38.8 | 3.23 | -180 | 185.6 | 95.0 | 81.6 | 65.1 | 47.2 | 30.2 | 11.10 |
| C1-12-CT-U | 121 | 73.3 | 1.18 | -202 | 28.8 | 45.1 | 37.5 | 30.3 | 22.1 | 14.1 | 5.27 |
| C1-12-CT-L | 121 | 70.9 | 1.09 | -199 | NA | 41.2 | 34.4 | 27.5 | 20.1 | 12.9 | 4.74 |
| | | | | | | | | | | | |
| C1-8-CapW | | | | | | | | | | | |
| C1-8-MFT-U | 153 | 40.6 | 3.05 | -157 | | 96.0 | 83.9 | 68.0 | 49.9 | 31.4 | 10.70 |
| C1-8-MFT-L | 153 | 40.6 | 3.34 | -163 | | 91.8 | 79.6 | 64.1 | 46.5 | 29.1 | 10.10 |
| C1-8-CT-U | 153 | 73.1 | 1.40 | -197 | | 35.8 | 29.7 | 23.5 | 17.3 | 11.3 | 4.25 |
| C1-8-CT-L | 153 | 75.0 | 1.33 | -183 | | 41.9 | 35.1 | 27.8 | 20.2 | 12.8 | 4.52 |

**Table B-9.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | AVS (mg/kg of dry sample) | Particle Size Distribution (% of solids less than) | | | | | |
| | | | | | | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| C1-9-CapW | 182 | | | | | | | | | | |
| C1-9-MFT-U | 182 | 40.1 | 3.52 | -162 | | 84.7 | 71.9 | 55.9 | 39.2 | 24.6 | 8.59 |
| C1-9-MFT-L | 182 | 41.3 | 3.34 | -179 | | 88.6 | 76.3 | 60.5 | 43.4 | 27.2 | 9.41 |
| C1-9-CT-U | 182 | 73.1 | 1.43 | -202 | | 28.7 | 23.1 | 17.7 | 12.5 | 8.0 | 3.14 |
| C1-9-CT-L | 182 | 73.8 | 1.32 | -199 | | 45.2 | 37.9 | 30.0 | 21.7 | 13.7 | 4.86 |
| | | | | | | | | | | | |
| C1-10-CapW | 213 | | | | | | | | | | |
| C1-10-MFT-U | 213 | 40.8 | 3.39 | -76 | | 92 | 80.4 | 64.7 | 46.5 | 29.2 | 10.90 |
| C1-10-MFT-L | 213 | 39.9 | 3.37 | -182 | | 91 | 78.4 | 62.5 | 45.6 | 30.0 | 12.10 |
| C1-10-CT-U | 213 | 74.8 | 1.49 | -206 | | 31 | 25.8 | 20.3 | 14.5 | 9.5 | 4.01 |
| C1-10-CT-L | 213 | 76.6 | 1.45 | -195 | | 34 | 28.7 | 22.9 | 16.8 | 11.3 | 4.76 |
| | | | | | | | | | | | |
| C1-11-CapW | 244 | | | | | | | | | | |
| C1-11-MFT-U | 244 | 40.4 | 3.39 | -152.2 | | 89 | 76.1 | 59.9 | 42.4 | 26.5 | 10.50 |
| C1-11-MFT-L | 244 | 40.5 | 3.49 | -125.9 | | 92 | 79.6 | 64.0 | 46.5 | 30.0 | 12.20 |
| C1-11-CT-U | 244 | 73.4 | 1.46 | -193.6 | | 40 | 33.1 | 26.2 | 19.0 | 12.4 | 5.30 |
| C1-11-CT-L | 244 | 74.6 | 1.24 | -187.6 | | 37 | 31.9 | 25.9 | 19.3 | 12.6 | 5.02 |
| | | | | | | | | | | | |
| C1-13-CapW | 267 | | | | | | | | | | |
| C1-13-MFT-U | 267 | 40.0 | 3.31 | -161.3 | | 92 | 78.2 | 63.2 | 41.0 | 23.1 | 8.14 |
| C1-13-MFT-L | 267 | 40.4 | 3.42 | -178.9 | | 90 | 76.9 | 60.2 | 42.5 | 26.9 | 8.11 |
| C1-13-CT-U | 267 | 71.0 | 1.01 | -196.6 | | 31 | 25.2 | 20.1 | 14.2 | 8.4 | 2.33 |
| C1-13-CT-L | 267 | 72.8 | 1.24 | -196.3 | | 28 | 23.9 | 19.2 | 14.0 | 9.0 | 2.77 |

**Table B-9.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | AVS (mg/kg of dry sample) | Particle Size Distribution (% of solids less than) | | | | | |
| | | | | | | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| C1-14-CapW | 303 | | | | | | | | | | |
| C1-14-MFT-U | 303 | 41.9 | 2.61 | -151 | | 91.7 | 79.6 | 64.1 | 45.7 | 27.9 | 10.2 |
| C1-14-MFT-L | 303 | 41.2 | 3.60 | -121 | | 95.8 | 83.1 | 66.2 | 48.6 | 32.5 | 13.3 |
| C1-14-CT-U | 303 | 72.9 | 0.73 | -192 | | 34.7 | 29.4 | 23.8 | 16.7 | 9.8 | 3.7 |
| C1-14-CT-L | 303 | 73.7 | 1.04 | -192 | | 82.7 | 69.7 | 56.7 | 42.0 | 26.4 | 9.5 |
| | | | | | | | | | | | |
| C1-15-CapW | 335 | | | | | | | | | | |
| C1-15-MFT-U | 335 | 43.0 | 3.70 | -121.9 | | 88.4 | 76.0 | 60.2 | 43.1 | 27.1 | 9.69 |
| C1-15-MFT-L | 335 | 43.9 | 3.84 | -149.8 | | 87.6 | 74.8 | 58.7 | 42.0 | 26.9 | 10.10 |
| C1-15-CT-U | 335 | 75.6 | 1.55 | -186.4 | | 28.5 | 23.4 | 18.2 | 13.1 | 8.6 | 3.49 |
| C1-15-CT-L | 335 | 76.3 | 1.34 | -188.3 | | 34.3 | 28.6 | 22.7 | 16.5 | 10.5 | 3.83 |
| | | | | | | | | | | | |
| C1-16-CapW | 366 | | | | | | | | | | |
| C1-16-MFT-U | 366 | 42.5 | 3.77 | -147 | | 90.9 | 77.9 | 61.4 | 44.3 | 28.7 | 11.30 |
| C1-16-MFT-L | 366 | 42.9 | 3.67 | -164 | | 91.8 | 79.1 | 62.7 | 45.5 | 29.5 | 11.40 |
| C1-16-CT-U | 366 | 73.2 | 1.19 | -178 | | 46.9 | 39.8 | 32.1 | 22.8 | 13.5 | 4.62 |
| C1-16-CT-L | 366 | 73.5 | 1.47 | -181 | | 34.8 | 28.9 | 22.7 | 16.5 | 10.9 | 4.47 |

1). NA=not available.
2). BDL=below detection limits.

**Table B-10. Physical and chemical analyses in System 2.**

| | | WATER PROPERTIES | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | |
| Sample | $t_{incu}$ (d) | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C2-1-CapW(1) | 1 | 7.9 | 4850 | 39 | 11 | BDL | 1070 | 17 | 18.9 | 28 | BDL | 910 | 212 | 1340 | BDL | 3.6 | 0.5 | BDL | 4.37 | 1.13 |
| C2-1-CapW(2) | 3 | 8 | 5130 | 64 | NA | BDL | 1150 | 17 | 21.1 | 32 | 7.7 | 940 | 326 | 1390 | BDL | 3.5 | 0.5 | BDL | 4.74 | 1.27 |
| C2-1-CapW(3) | 7 | 8 | 5130 | 63 | 10.7 | BDL | 1150 | 17 | 20.6 | 31 | BDL | 930 | 333 | 1220 | BDL | 3.5 | 0.5 | BDL | 4.82 | 1.25 |
| C2-1-CapW(4) | 14 | 8.1 | 5170 | 72 | NA | BDL | 1110 | 17 | 20.8 | 32 | 11 | 940 | 346 | 1230 | BDL | 2.2 | 0.5 | BDL | 4.9 | 1.31 |
| C2-1-CapW(5) | 21 | 8.1 | 5070 | 69 | 9.8 | BDL | 1110 | 17 | 19 | 28 | 8.4 | 940 | 252 | 1050 | BDL | 2.1 | 0.5 | BDL | 4.93 | 1.19 |
| C2-1-CapW(6) | 35 | 8 | 5140 | 85 | NA | BDL | 1150 | 16 | 19.3 | 29 | BDL | 930 | 213 | 1340 | BDL | 3.2 | 0.5 | BDL | 4.94 | 1.2 |
| C2-1-CapW(7) | 49 | 8.1 | 4940 | 100 | 8 | BDL | 1070 | 15 | 19.3 | 28 | BDL | 920 | 199 | NA | BDL | 3.2 | 0.5 | BDL | 4.61 | 1.2 |
| C2-1-CapW(8) | 77 | 8 | 5330 | 120 | 8.13 | BDL | 1170 | 16 | 19.6 | 27 | BDL | 930 | 255 | 1530 | BDL | 3.2 | 0.5 | BDL | 4.56 | 1.17 |
| C2-1-CapW(9) | 133 | 8.1 | 5230 | 150 | 8.42 | BDL | 1090 | 15 | 18.9 | 26 | BDL | 940 | 423 | 1494 | BDL | 2.9 | 0.4 | BDL | 4.05 | 1.14 |
| C2-1-CapW(10) | 352 | 8.2 | 5520 | 160 | 0.4 | BDL | 1270 | 25.0 | 19.1 | 25.2 | BDL | 920 | 593 | 1240 | BDL | 2.98 | 0.13 | BDL | 4.52 | 1.17 |
| C2-1-MFT-U | 352 | 8.1 | 5010 | 180 | 11.0 | BDL | 1170 | 14.9 | 11.8 | 13.3 | BDL | 970 | 96 | 1360 | BDL | 2.62 | 0.24 | BDL | 3.76 | 0.64 |
| C2-1-MFT-L | 352 | 8 | 4950 | 190 | 11.0 | BDL | 1120 | 13.4 | 11.5 | 14.8 | BDL | 980 | 110 | 1320 | BDL | 2.70 | 0.27 | BDL | 3.97 | 0.65 |
| C2-1-CT-U | 352 | 8.1 | 5100 | 210 | 12.0 | BDL | 1180 | 13.6 | 11.6 | 21.2 | BDL | 1000 | 129 | 1380 | BDL | 3.03 | 0.39 | BDL | 4.55 | 0.69 |
| C2-1-CT-L | 352 | 8 | 5200 | 220 | 10.0 | BDL | 1180 | 14.9 | 12.4 | 24.3 | BDL | 1000 | 138 | 1250 | BDL | 3.31 | 0.44 | BDL | 5.09 | 0.76 |

**Table B-10.** *(continued).*

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C2-2-CapW(1) | 1 | 7.9 | 4760 | 27 | 10.8 | BDL | 1100 | 17 | 18.4 | 27 | BDL | 880 | 157 | 1130 | BDL | 3.6 | 0.5 | BDL | 4.32 | 1.1 |
| C2-2-CapW(2) | 3 | 8 | 4960 | 49 | NA | BDL | 1130 | 17 | 19.6 | 29 | BDL | 910 | 243 | 1010 | BDL | 3.5 | 0.5 | BDL | 4.64 | 1.18 |
| C2-2-CapW(3) | 7 | 8 | 4980 | 55 | 10.1 | BDL | 1130 | 17 | 19.2 | 29 | BDL | 900 | 261 | 860 | BDL | 3.5 | 0.5 | BDL | 4.56 | 1.16 |
| C2-2-CapW(4) | 14 | 8.2 | 4920 | 59 | NA | BDL | 1070 | 17 | 18.7 | 27 | BDL | 890 | 216 | 1120 | BDL | 2.1 | 0.5 | BDL | 4.64 | 1.16 |
| C2-2-CapW(5) | 21 | 8.2 | 4930 | 69 | 9.2 | BDL | 1060 | 16 | 17.4 | 25 | BDL | 940 | 168 | 1220 | BDL | 2 | 0.5 | BDL | 4.85 | 1.07 |
| C2-2-CapW(6) | 35 | 8.1 | 4930 | 78 | NA | BDL | 1110 | 16 | 17.4 | 25 | BDL | 940 | 146 | 1070 | BDL | 3.3 | 0.5 | BDL | 4.85 | 1.06 |
| C2-2-CapW(7) | 49 | 8.2 | 4660 | 82 | 7.6 | BDL | 1010 | 14 | 16.4 | 22 | BDL | 870 | 128 | | BDL | 3.3 | 0.4 | BDL | 4.57 | 1 |
| C2-2-CapW(8) | 77 | 8.1 | 4970 | 110 | 8.33 | BDL | 1090 | 15 | 15.9 | 19 | BDL | 930 | 147 | 1390 | BDL | 3.3 | 0.3 | BDL | 4.52 | 0.93 |
| C2-2-CapW(9) | 133 | 8.3 | 4800 | 130 | 7.07 | BDL | 995 | 14 | 14.9 | 17 | BDL | 910 | 278 | 1356 | BDL | 3 | 0.3 | BDL | 4.06 | 0.87 |
| | | | | | | | | | | | | | | | | | | | | |
| C2-3-CapW(1) | 1 | 7.9 | 4890 | 32 | 11.2 | BDL | 1110 | 17 | 19.5 | 29 | BDL | 890 | 202 | 1140 | BDL | 3.6 | 0.5 | BDL | 4.37 | 1.19 |
| C2-3-CapW(2) | 3 | 8 | 5060 | 57 | NA | BDL | 1160 | 17 | 21 | 32 | BDL | 910 | 324 | 1150 | BDL | 3.5 | 0.6 | BDL | 4.54 | 1.28 |
| C2-3-CapW(3) | 7 | 8 | 5140 | 64 | 10.6 | BDL | 1150 | 17 | 21 | 32 | BDL | 920 | 360 | 1080 | BDL | 3.5 | 0.5 | BDL | 4.68 | 1.27 |
| C2-3-CapW(4) | 14 | 8 | 5020 | 58 | NA | BDL | 1060 | 16 | 19.1 | 29 | BDL | 920 | 263 | 1420 | BDL | 2.1 | 0.5 | BDL | 4.78 | 1.2 |
| C2-3-CapW(5) | 21 | 8.1 | 5060 | 69 | 9.9 | BDL | 1070 | 16 | 18.7 | 28 | BDL | 910 | 254 | 1430 | BDL | 1.9 | 0.5 | BDL | 4.74 | 1.17 |
| C2-3-CapW(6) | 35 | 8 | 5090 | 79 | NA | BDL | 1160 | 16 | 18.9 | 29 | BDL | 940 | 145 | 1080 | BDL | 3.3 | 0.5 | BDL | 5.02 | 1.18 |
| C2-3-CapW(7) | 49 | 8.1 | 4780 | 90 | 7.8 | BDL | 1010 | 15 | 17.7 | 27 | BDL | 890 | 135 | NA | BDL | 3.2 | 0.5 | BDL | 4.67 | 1.1 |
| C2-3-CapW(8) | 77 | 8 | 5150 | 110 | 8.2 | BDL | 1140 | 16 | 18.6 | 28 | 5 | 930 | 170 | 1510 | BDL | 3.3 | 0.5 | BDL | 4.68 | 1.12 |
| C2-3-CapW(9) | 133 | 8.1 | 5090 | 140 | 8.9 | BDL | 1070 | 15 | 18.5 | 28 | BDL | 930 | 346 | 1464 | BDL | 3 | 0.4 | BDL | 4.26 | 1.14 |

**Table B-10.** *(continued).*

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | |
| | | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C2-4-CapW(1) | 1 | 7.9 | 5290 | 69 | 11.4 | BDL | 1160 | 18 | 23.7 | 38 | BDL | 910 | 476 | 884 | BDL | 3.33 | 0.6 | BDL | 4.1 | 1.45 |
| C2-4-CapW(2) | 3 | 7.9 | 5480 | 79 | NA | BDL | 1200 | 18 | 24.1 | 40 | BDL | 930 | 560 | 1120 | BDL | 3.29 | 0.5 | BDL | 4.4 | 1.47 |
| C2-4-CapW(3) | 7 | 7.9 | 5330 | 67 | 10.6 | BDL | 1170 | 17 | 22.4 | 37 | BDL | 910 | 487 | 1300 | BDL | 3.39 | 0.5 | BDL | 4.6 | 1.36 |
| C2-4-CapW(4) | 14 | 8 | 5230 | 66 | NA | BDL | 1130 | 17 | 21.5 | 34 | BDL | 930 | 392 | 1030 | BDL | 2.11 | 0.5 | BDL | 4.8 | 1.35 |
| C2-4-CapW(5) | 21 | 8.1 | 5200 | 74 | 9.8 | BDL | 1100 | 17 | 19.8 | 30 | BDL | 930 | 333 | 1160 | BDL | 1.93 | 0.5 | BDL | 4.7 | 1.24 |
| C2-4-CapW(6) | 35 | 7.9 | 5260 | 85 | NA | BDL | 1200 | 17 | 20.4 | 31 | BDL | 940 | 160 | 1450 | BDL | 3.32 | 0.5 | BDL | 5.1 | 1.28 |
| C2-4-CapW(7) | 49 | 8.1 | 4970 | 97 | 8.4 | BDL | 1050 | 15 | 19.4 | 30 | BDL | 880 | 158 | NA | BDL | 3.18 | 0.5 | BDL | 4.7 | 1.2 |
| C2-4-CapW(8) | 77 | 8 | 5370 | 120 | 8.71 | BDL | 1160 | 16 | 19.9 | 30 | BDL | 940 | 198 | 1660 | BDL | 3.26 | 0.5 | BDL | 4.6 | 1.2 |
| C2-4-CapW(9) | 133 | 8 | 5340 | 150 | 9.56 | BDL | 1110 | 15 | 20.3 | 31 | BDL | 930 | 411 | 1526 | BDL | 2.97 | 0.5 | BDL | 4.3 | 1.25 |
| C2-4-CapW(10) | 352 | 8 | 5920 | 190 | 9.1 | BDL | 1350 | 17.0 | 22.3 | 30.9 | BDL | 970 | 590 | 1430 | BDL | 3.08 | 0.22 | BDL | 4.30 | 1.40 |
| C2-4-MFT-U | 352 | 8 | 5250 | 210 | 7.9 | BDL | 1200 | 12.8 | 12.4 | 12.9 | BDL | 1000 | 107 | 1390 | BDL | 2.66 | 0.25 | BDL | 3.63 | 0.65 |
| C2-4-MFT-L | 352 | 7.8 | 5210 | 210 | 8.6 | BDL | 1200 | 12.2 | 11.9 | 14.2 | BDL | 1000 | 161 | 1280 | BDL | 2.74 | 0.27 | BDL | 3.77 | 0.65 |
| C2-4-CT-U | 352 | 7.8 | 5440 | 240 | 6.9 | BDL | 1300 | 13.1 | 12.0 | 20.6 | BDL | 1000 | 277 | 1440 | BDL | 3.10 | 0.42 | BDL | 4.31 | 0.69 |
| C2-4-CT-L | 352 | 8 | 5560 | 260 | 7.3 | BDL | 1400 | 14.6 | 13.2 | 24.7 | BDL | 1100 | 329 | 1240 | BDL | 3.48 | 0.49 | BDL | 5.06 | 0.77 |

**Table B-10.** *(continued).*

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | pH | EC (uS/cm) | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | | | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C2-5-CapW(1) | 1 | 7.9 | 5130 | 33 | 11.2 | BDL | 1150 | 18 | 22.5 | 33 | BDL | 890 | 431 | 1010 | BDL | 3.4 | 0.57 | BDL | 4.3 | 1.36 |
| C2-5-CapW(2) | 3 | 8.15 | 5650 | 70 | NA | BDL | 1230 | 18 | 25.9 | 40 | BDL | 900 | 700 | 1040 | BDL | 3.3 | 0.29 | BDL | 4.3 | 1.54 |
| C2-5-CapW(3) | 7 | 8.03 | 5690 | 76 | 11.4 | BDL | 1280 | 18 | 25.7 | 41 | BDL | 910 | 690 | 964 | BDL | 3.4 | 0.34 | BDL | 4.5 | 1.55 |
| C2-5-CapW(4) | 14 | 8 | 5690 | 87 | NA | BDL | 1200 | 18 | 25.2 | 40 | BDL | 920 | 676 | 904 | BDL | 1.9 | 0.16 | BDL | 4.6 | 1.57 |
| C2-5-CapW(5) | 21 | 8.05 | 5690 | 98 | 10.1 | BDL | 1170 | 17 | 23.5 | 36 | BDL | 940 | 627 | 1330 | BDL | 1.7 | 0.28 | BDL | 4.6 | 1.45 |
| C2-5-CapW(6) | 35 | 8 | 5630 | 110 | NA | BDL | 1280 | 18 | 23.3 | 36 | BDL | 940 | 461 | 1780 | BDL | 3.2 | 0.29 | BDL | 4.9 | 1.44 |
| C2-5-CapW(7) | 49 | 7.98 | 5320 | 120 | 8.7 | BDL | 1170 | 16 | 22.3 | 32 | BDL | 980 | 380 | NA | BDL | 3.1 | 0.31 | BDL | 4.6 | 1.35 |
| C2-5-CapW(8) | 77 | 8 | 5620 | 130 | 8.77 | BDL | 1230 | 16 | 21.4 | 27 | BDL | 940 | 383 | 1400 | BDL | 3.2 | 0.27 | BDL | 4.5 | 1.2 |
| C2-5-CapW(9) | 133 | 8.12 | 5460 | 150 | 9.25 | BDL | 1140 | 15 | 20.5 | 26 | BDL | 920 | 438 | 1606 | BDL | 2.9 | 0.27 | BDL | 4 | 1.18 |

**Table B-10.** *(continued).*

| Sample | $t_{incu}$ (d) | WATER PROPERTIES | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | (mg/L) | | | Main Ions (mg/L) | | | | | | | | Minor Elements (mg/L) | | | | | |
| | | pH | EC (uS/cm) | $Br^-$ | $N(NH_4^+)$ | $S^{2-}$ | $Na^+$ | $K^+$ | $Mg^{2+}$ | $Ca^{2+}$ | $F^-$ | $Cl^-$ | $SO_4^{2-}$ | $HCO_3^-$ | Al | B | Ba | Fe | Si | Sr |
| C2-6-CapW(1) | 1 | 7.93 | 4770 | 8.9 | 10.7 | BDL | 1060 | 16 | 18.5 | 27 | BDL | 870 | 142 | 1410 | BDL | 3.6 | 0.48 | BDL | 4.5 | 1.1 |
| C2-6-CapW(2) | 3 | 8.01 | 4950 | 23 | NA | BDL | 1110 | 17 | 19.8 | 29 | BDL | 880 | 216 | 1550 | BDL | 3.6 | 0.51 | BDL | 4.8 | 1.2 |
| C2-6-CapW(3) | 7 | 7.95 | 5340 | 47 | 10.9 | BDL | 1130 | 17 | 21.8 | 32 | BDL | 900 | 388 | 1520 | BDL | 3.3 | 0.57 | BDL | 4.6 | 1.3 |
| C2-6-CapW(4) | 14 | 8.01 | 5320 | 56 | NA | BDL | 1120 | 17 | 21.5 | 32 | BDL | 900 | 365 | 1300 | BDL | 1.9 | 0.59 | BDL | 4.7 | 1.3 |
| C2-6-CapW(5) | 21 | 8.05 | 5490 | 75 | 10.1 | BDL | 1200 | 18 | 22.2 | 30 | BDL | 930 | 367 | 1200 | BDL | 1.9 | 0.58 | BDL | 4.7 | 1.4 |
| C2-6-CapW(6) | 35 | 8 | 5670 | 96 | NA | BDL | 1300 | 18 | 23 | 33 | BDL | 930 | 320 | 1520 | BDL | 3.1 | 0.59 | BDL | 4.7 | 1.4 |
| C2-6-CapW(7) | 49 | 8.05 | 5450 | 110 | 9.5 | BDL | 1160 | 16 | 23 | 30 | BDL | 910 | 289 | NA | BDL | 2.9 | 0.56 | BDL | 4.3 | 1.4 |
| C2-6-CapW(8) | 77 | 8 | 5930 | 140 | 10.7 | BDL | 1310 | 17 | 23.5 | 26 | BDL | 940 | 284 | 1820 | BDL | 3 | 0.49 | BDL | 4.4 | 1.3 |
| C2-6-CapW(9) | 133 | 7.98 | 6020 | 170 | 10.4 | BDL | 1280 | 17 | 25.2 | 35 | BDL | 950 | 583 | 1858 | BDL | 2.8 | 0.23 | BDL | 4.1 | 1.5 |
| C2-6-CapW(10) | 352 | 7.88 | 6800 | 200 | 13.0 | BDL | 1660 | 18.8 | 27.4 | 30.6 | BDL | 970 | 868 | 1750 | BDL | 3.09 | 0.13 | BDL | 4.37 | 1.64 |
| C2-6-MFT-U | 352 | 7.88 | 6000 | 220 | 8.3 | BDL | 1450 | 14.2 | 14.3 | 12.5 | BDL | 1000 | 282 | 1770 | BDL | 2.81 | 0.24 | BDL | 3.48 | 0.70 |
| C2-6-MFT-L | 352 | 7.91 | 5960 | 220 | 7.6 | BDL | 1440 | 12.7 | 12.3 | 11.1 | BDL | 1000 | 288 | 1690 | BDL | 2.72 | 0.25 | BDL | 3.42 | 0.60 |
| C2-6-CT-U | 352 | 7.95 | 6530 | 250 | 7.1 | BDL | 1560 | 13.4 | 11.7 | 12.8 | BDL | 1000 | 532 | 1850 | BDL | 2.76 | 0.25 | BDL | 3.86 | 0.58 |
| C2-6-CT-L | 352 | 8.07 | 6620 | 260 | 7.0 | BDL | 1590 | 13.3 | 12.0 | 15.2 | BDL | 1000 | 335 | 2410 | BDL | 2.85 | 0.11 | BDL | 4.05 | 0.57 |

**Table B-10.** *(continued).*

| Sample | $t_{incu}$ (d) | SOLIDS PROPERTIES | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Particle Size Distribution (% of solids less than) | | | | | |
| | | Solids content (g/100g) | Oil content (g/100g) | Eh (mV) | 44 um | 22 um | 11 um | 5.5 um | 2.8 um | 1.0 um |
| C2-1-CapW(10) | 352 | | | | | | | | | |
| C2-1-MFT-U | 352 | 43.0 | 3.49 | -167 | 91.5 | 78.8 | 62.8 | 45.8 | 29.2 | 10.6 |
| C2-1-MFT-L | 352 | 45.5 | 3.67 | -180 | 90.2 | 77.9 | 62.5 | 45.3 | 28.1 | 9.7 |
| C2-1-CT-U | 352 | 76.1 | 1.26 | -187 | 39.7 | 33.6 | 27.1 | 19.7 | 12.3 | 4.4 |
| C2-1-CT-L | 352 | 74.5 | 0.96 | -186 | 43.3 | 37.2 | 30.6 | 21.5 | 12.2 | 4.2 |
| C2-4-CapW(10) | 352 | | | | | | | | | |
| C2-4-MFT-U | 352 | 42.9 | 3.70 | -179 | 91.6 | 78.6 | 62.3 | 45.2 | 29.1 | 10.9 |
| C2-4-MFT-L | 352 | 43.8 | 3.52 | -186 | 90.2 | 77.8 | 62.4 | 44.9 | 27.7 | 9.6 |
| C2-4-CT-U | 352 | 74.9 | 1.11 | -196 | 38.9 | 33.0 | 26.9 | 19.2 | 11.3 | 3.9 |
| C2-4-CT-L | 352 | 75.2 | 1.51 | -203 | 37.9 | 31.6 | 25.0 | 18.2 | 11.9 | 4.7 |
| C2-6-CapW(10) | 352 | | | | | | | | | |
| C2-6-MFT-U | 352 | 41.4 | 3.34 | -197 | 90.5 | 77.9 | 62.1 | 44.8 | 28.2 | 10.2 |
| C2-6-MFT-L | 352 | 44.1 | 3.51 | -199 | 91.9 | 79.1 | 62.8 | 45.6 | 29.1 | 10.8 |
| C2-6-CT-U | 352 | 75.3 | 0.31 | -215 | 46.0 | 40.0 | 32.8 | 21.8 | 11.7 | 4.3 |
| C2-6-CT-L | 352 | 74.5 | 1.36 | -214 | 38.2 | 31.8 | 25.1 | 18.3 | 11.8 | 4.5 |

1). NA=not available.
2). BDL=below detection limits.

**Table B-11. Trapped gas analyses on the baseline MFT and CT samples.**

| Sample | $t_{incu}$ (d) | Trapped gas analysis (vol%) | | | |
|---|---|---|---|---|---|
| | | $CH_4$ | $CO_2$ | $C_2H_4$ | $H_2S$ |
| Base MFT 1 | 0 | 3.48 | 0.988 | nd | nd |
| Base MFT 2 | 0 | 2.85 | 1.14 | nd | nd |
| Base MFT 3 | 0 | 0.046 | 0.168 | 0.027 | nd |
| Base CT 1 | 0 | 0.072 | 0.180 | nd | nd |
| Base CT 2 | 0 | 0.053 | 0.211 | nd | nd |
| Base CT 3 | 0 | 0.126 | 0.269 | nd | nd |
| Base CT 4 | 0 | nd | 0.158 | nd | nd |
| Base CT 5 | 0 | nd | 0.194 | 0.016 | nd |
| Base CT 6 | 0 | nd | 0.066 | 0.090 | nd |

1). Using Micro-GC and the GC parameters at Table 4-2 with 30 s of sampling time.
2). nd=non-detectable.

**Table B-12. Trapped gas analyses on MFT and CT samples in Systems 1, 2, 3, and 4.**

| Sample | $t_{incu}$ (d) | Trapped gas analysis (vol%) | | | |
|---|---|---|---|---|---|
| | | $CH_4$ | $CO_2$ | $C_2H_4$ | $H_2S$ |
| **System 1 (Cap water/MFT/CT)** | | | | | |
| C1-4-MFT-U | 29 | 0.028 | 0.202 | nd | nd |
| C1-4-MFT-L | 29 | 0.031 | 0.130 | nd | nd |
| C1-4-CT-U | 29 | nd | 0.007 | 0.006 | nd |
| C1-4-CT-L | 29 | nd | 0.205 | nd | nd |
| C1-5-MFT-U | 62 | 0.386 | 0.135 | nd | nd |
| C1-5-MFT-L | 62 | 0.289 | 0.078 | 0.076 | nd |
| C1-5-CT-U | 62 | nd | 0.105 | 0.077 | 0.418 |
| C1-5-CT-L | 62 | 0.050 | 0.102 | nd | nd |
| C1-6-MFT-U | 91 | 0.082 | 0.383 | nd | nd |
| C1-6-MFT-L | 91 | 0.683 | 0.087 | 0.097 | nd |
| C1-6-CT-U | 91 | 0.150 | 0.066 | nd | nd |
| C1-6-CT-L | 91 | 0.069 | 0.057 | 0.094 | 0.246 |
| C1-12-MFT-U | 121 | 0.067 | 0.266 | nd | nd |
| C1-12-MFT-L | 121 | 0.248 | 0.263 | 0.033 | nd |
| C1-12-CT-U | 121 | 0.101 | 0.256 | 0.117 | 0.502 |
| C1-12-CT-L | 121 | 0.205 | 0.170 | 0.024 | 0.009 |
| C1-8-MFT-U | 153 | 0.367 | 0.390 | nd | nd |
| C1-8-MFT-L | 153 | 0.505 | 0.116 | nd | 0.037 |
| C1-8-CT-U | 153 | 0.169 | 0.320 | 0.035 | 0.080 |
| C1-8-CT-L | 153 | 0.094 | 0.332 | nd | 0.020 |
| C1-9-MFT-U | 182 | 0.182 | 0.761 | 0.577 | 0.097 |
| C1-9-MFT-L | 182 | 0.074 | 0.172 | nd | nd |
| C1-9-CT-U | 182 | nd | 0.390 | nd | 0.035 |
| C1-9-CT-L | 182 | 0.082 | 0.400 | 0.110 | 0.028 |
| C1-10-MFT-U | 213 | 0.053 | 0.269 | nd | |
| C1-10-MFT-L | 213 | 0.098 | 0.036 | nd | 0.425 |
| C1-10-CT-U | 213 | nd | 0.140 | nd | 0.138 |
| C1-10-CT-L | 213 | nd | 0.121 | nd | nd |
| C1-11-MFT-U | 244 | 0.048 | 0.0811 | nd | nd |
| C1-11-MFT-L | 244 | 0.072 | 0.0932 | nd | nd |
| C1-11-CT-U | 244 | 0.049 | 0.0806 | nd | nd |
| C1-11-CT-L | 244 | 0.034 | 0.0880 | nd | 0.050 |
| C1-13-MFT-U | 267 | 0.061 | 0.063 | nd | 0.012 |
| C1-13-MFT-L | 267 | 0.091 | 0.076 | nd | 0.036 |
| C1-13-CT-U | 267 | nd | 0.086 | nd | nd |
| C1-13-CT-L | 267 | 0.045 | 0.088 | nd | 0.054 |

**Table B-12.** *(continued).*

| Sample | $t_{incu}$ (d) | Trapped gas analysis (vol%) | | | |
|---|---|---|---|---|---|
| | | $CH_4$ | $CO_2$ | $C_2H_4$ | $H_2S$ |
| **System 1 (Cap water/MFT/CT)** *(continued)* | | | | | |
| C1-14-MFT-U | 303 | 0.050 | 0.083 | nd | nd |
| C1-14-MFT-L | 303 | 0.036 | 0.118 | nd | nd |
| C1-14-CT-U | 303 | 0.310 | 0.115 | nd | nd |
| C1-14-CT-L | 303 | 0.075 | 0.118 | nd | nd |
| C1-15-MFT-U | 335 | 0.257 | 0.070 | nd | nd |
| C1-15-MFT-L | 335 | 0.222 | 0.074 | nd | nd |
| C1-15-CT-U | 335 | 0.148 | 0.079 | nd | nd |
| C1-15-CT-L | 335 | 0.042 | 0.072 | nd | nd |
| C1-16-MFT-U | 366 | 1.13 | 1.39 | nd | nd |
| C1-16-MFT-L | 366 | 0.752 | 0.120 | nd | nd |
| C1-16-CT-U | 366 | 0.482 | 0.175 | nd | nd |
| C1-16-CT-L | 366 | 0.412 | 0.115 | nd | nd |
| **System 2 (Cap water/MFT/CT)** | | | | | |
| C2-1-MFT-U | 352 | 1.04 | 0.090 | nd | nd |
| C2-1-MFT-L | 352 | 1.64 | 0.114 | nd | nd |
| C2-1-CT-U | 352 | 1.02 | 0.064 | nd | nd |
| C2-1-CT-L | 352 | 0.674 | 0.060 | nd | nd |
| C2-4-MFT-U | 352 | 1.66 | 0.161 | nd | nd |
| C2-4-MFT-L | 352 | 1.71 | 0.149 | nd | nd |
| C2-4-CT-U | 352 | 0.730 | 0.092 | nd | nd |
| C2-4-CT-L | 352 | 0.228 | 0.075 | nd | nd |
| C2-6-MFT-U | 352 | 1.12 | 0.148 | nd | nd |
| C2-6-MFT-L | 352 | 1.62 | 0.108 | nd | nd |
| C2-6-CT-U | 352 | 1.08 | 0.057 | nd | nd |
| C2-6-CT-L | 352 | 0.718 | 0.043 | nd | nd |

**Table B-12.** *(continued)*

| Sample | $t_{incu}$ (d) | Trapped gas analysis (vol%) | | | |
|---|---|---|---|---|---|
| | | $CH_4$ | $CO_2$ | $C_2H_4$ | $H_2S$ |
| **System 3 (Cap water/MFT)** | | | | | |
| C3-1-MFT-U | 28 | 0.383 | 0.196 | nd | nd |
| C3-1-MFT-L | 28 | 0.139 | 0.154 | 0.049 | nd |
| C3-2-MFT-U | 61 | 0.857 | 0.104 | nd | nd |
| C3-2-MFT-L | 61 | 1.06 | 0.122 | nd | 0.011 |
| C3-3-MFT-U | 90 | 0.457 | 0.164 | nd | nd |
| C3-3-MFT-L | 90 | 1.24 | 0.130 | 0.012 | nd |
| C3-4-MFT-U | 120 | 1.14 | 0.202 | 0.060 | nd |
| C3-4-MFT-L | 120 | 0.631 | 0.025 | 0.030 | 0.072 |
| C3-5-MFT-U | 152 | 0.399 | 0.291 | 0.357 | 0.102 |
| C3-5-MFT-L | 152 | 0.051 | 0.275 | 0.099 | nd |
| C3-6-MFT-U | 181 | 0.033 | 0.180 | nd | nd |
| C3-6-MFT-L | 181 | 0.149 | 0.524 | 0.119 | nd |
| C3-7-MFT-U | 212 | 0.058 | 0.284 | nd | nd |
| C3-7-MFT-L | 212 | 0.070 | 0.136 | 0.031 | 0.046 |
| C3-8-MFT-U | 243 | nd | 0.107 | nd | nd |
| C3-8-MFT-L | 243 | 0.101 | 0.117 | nd | nd |
| C3-9-MFT-U | 266 | 0.161 | 0.089 | nd | nd |
| C3-9-MFT-L | 266 | 0.150 | 0.103 | nd | 0.011 |
| C3-10-MFT-U | 302 | 0.138 | 0.143 | nd | nd |
| C3-10-MFT-L | 302 | 0.142 | 0.137 | nd | nd |
| C3-11-MFT-U | 334 | 0.585 | 0.129 | nd | nd |
| C3-11-MFT-L | 334 | 0.513 | 0.114 | nd | nd |
| C3-12-MFT-U | 365 | 0.233 | 0.129 | nd | nd |
| C3-12-MFT-L | 365 | nd | 0.603 | nd | nd |

**Table B-12.** *(continued).*

| Sample | $t_{incu}$ (d) | Trapped gas analysis (vol%) | | | |
|---|---|---|---|---|---|
| | | $CH_4$ | $CO_2$ | $C_2H_4$ | $H_2S$ |
| System 4 (Cap water/CT) | | | | | |
| C4-1-CT-U | 28 | nd | 0.021 | 0.023 | nd |
| C4-1-CT-L | 28 | 0.034 | 0.242 | nd | 0.062 |
| C4-2-CT-U | 61 | 0.037 | 0.277 | 0.255 | nd |
| C4-2-CT-L | 61 | nd | 0.133 | nd | nd |
| C4-3-CT-U | 90 | 0.031 | 0.311 | 0.071 | 0.010 |
| C4-3-CT-L | 90 | nd | 0.143 | 0.020 | nd |
| C4-4-CT-U | 120 | 0.135 | 0.082 | nd | nd |
| C4-4-CT-L | 120 | 0.277 | 0.093 | nd | nd |
| C4-5-CT-U | 152 | 0.085 | 0.387 | 0.090 | nd |
| C4-5-CT-L | 152 | nd | 0.152 | 0.025 | nd |
| C4-6-CT-U | 181 | nd | 0.343 | 0.089 | 0.018 |
| C4-6-CT-L | 181 | nd | 0.332 | 0.008 | nd |
| C4-7-CT-U | 212 | 0.078 | 0.293 | 0.193 | nd |
| C4-7-CT-L | 212 | nd | 0.316 | 0.052 | 1.10 |
| C4-8-CT-U | 243 | 0.069 | 0.086 | nd | nd |
| C4-8-CT-L | 243 | nd | 0.088 | nd | nd |
| C4-9-CT-U | 266 | nd | 0.104 | nd | nd |
| C4-9-CT-L | 266 | 0.041 | 0.103 | nd | nd |
| C4-10-CT-U | 302 | 0.051 | 0.127 | nd | nd |
| C4-10-CT-L | 302 | 0.047 | 0.125 | nd | nd |
| C4-11-CT-U | 334 | 0.084 | 0.068 | nd | nd |
| C4-11-CT-L | 334 | 0.195 | 0.053 | nd | nd |
| C4-12-CT-U | 365 | 1.12 | 0.165 | nd | nd |
| C4-12-CT-L | 365 | 0.171 | 0.369 | nd | nd |

1). Using Micro-GC and the GC parameters at Table 4-2 with 30 s sampling time.
2). nd=non-detectable.

**Table B-13. MPN analyses on MFT and CT samples of System 1.**

| Sample | $t_{incu}$ (d) | Methanogen MPN/g of dry solids | SRB MPN/g of dry solids |
|---|---|---|---|
| Base CT 1 | 0 | 1.57E+02 | 1.57E+05 |
| Base CT 2 | 0 | 1.40E+01 | 1.50E+06 |
| Base MFT 1 | 0 | 2.24E+03 | 1.97E+03 |
| C1-3-CT-M | 15 | 1.15E+03 | 6.80E+04 |
| C1-3-MFT-U | 15 | 5.04E+03 | 5.42E+05 |
| C1-3-MFT-L | 15 | 2.28E+03 | 4.83E+05 |
| C1-4-CT-M | 29 | 5.44E+03 | 3.09E+05 |
| C1-4-MFT-L | 29 | 5.75E+03 | 4.98E+05 |
| C1-4-MFT-U | 29 | 8.14E+03 | 2.00E+06 |
| C1-5-CT-M | 62 | 1.16E+03 | 1.22E+05 |
| C1-5-MFT-L | 62 | 1.95E+05 | 9.86E+05 |
| C1-5-MFT-U | 62 | 2.11E+05 | 5.09E+05 |
| C1-12-CT-M | 121 | 1.14E+04 | 2.84E+04 |
| C1-12-MFT-L | 121 | 5.22E+04 | 5.48E+05 |
| C1-12-MFT-U | 121 | 2.94E+03 | 8.46E+05 |
| C1-9-CT-M | 182 | 4.96E+03 | 2.75E+04 |
| C1-9-MFT-L | 182 | 4.67E+03 | 9.16E+05 |
| C1-9-MFT-U | 182 | 5.99E+05 | 1.08E+06 |
| C1-11-CT-M | 244 | 5.26E+04 | 2.81E+04 |
| C1-11-MFT-L | 244 | 8.62E+04 | 2.14E+06 |
| C1-11-MFT-U | 244 | 1.99E+05 | 5.36E+05 |
| C1-14-CT-M | 303 | 2.95E+04 | 2.51E+03 |
| C1-14-MFT-L | 303 | 5.18E+04 | 1.92E+06 |
| C1-14-MFT-U | 303 | 2.61E+05 | 2.34E+05 |
| C1-16-CT-M | 366 | 2.67E+04 | 9.76E+02 |
| C1-16-MFT-L | 366 | 4.96E+05 | 4.44E+06 |
| C1-16-MFT-U | 366 | 4.89E+05 | 4.09E+04 |

# Appendix C. Discussion

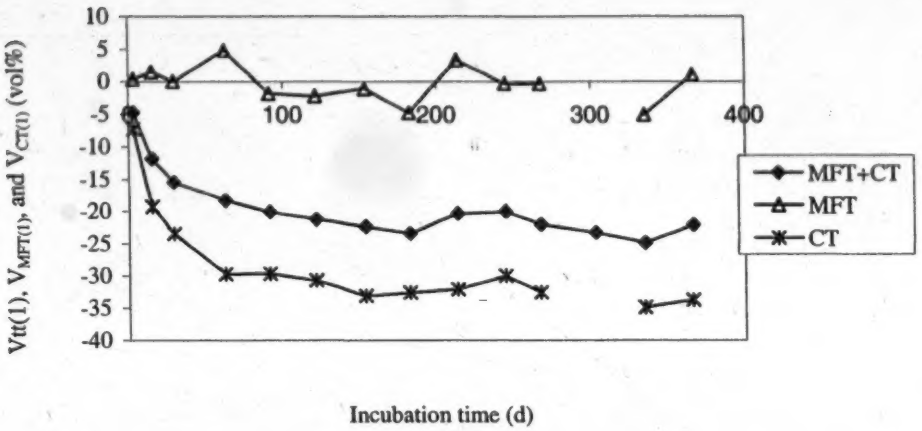



**Figure C-1. Total and individual tailings volume changes (Vtt, $V_{MFT}$, and $V_{CT}$) in System 1.**

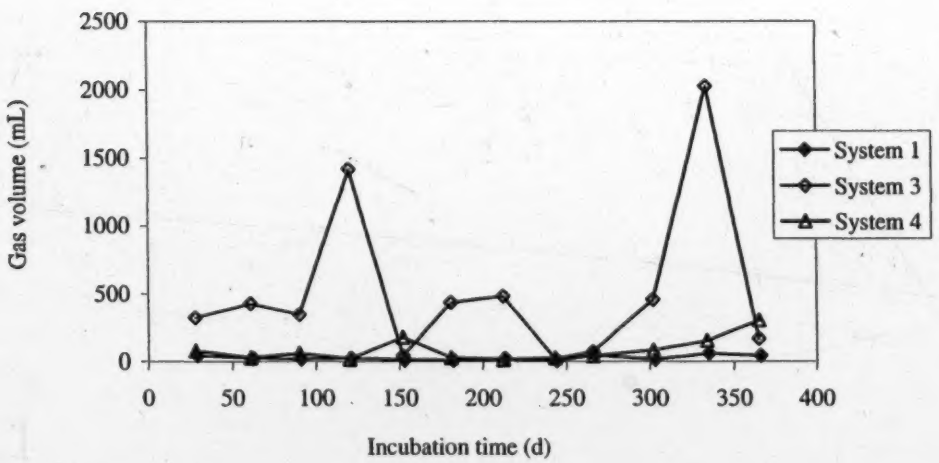



**Figure C-2. Tedlar bag gas volumes in Systems 1, 3, and 4.**

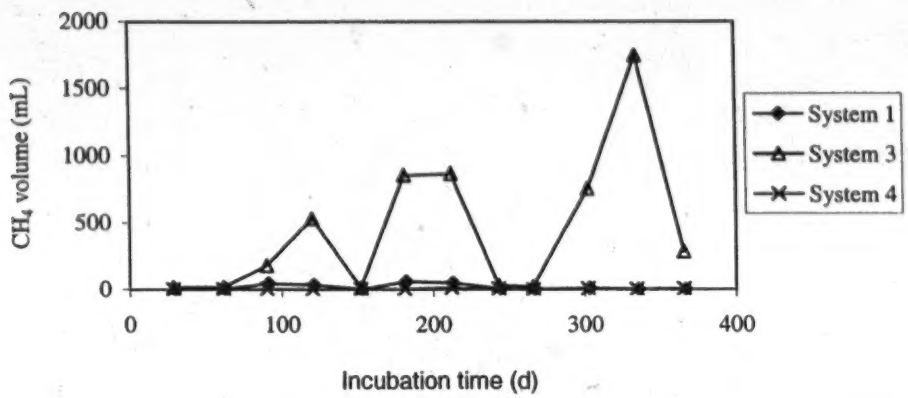



**Figure C-3. Total $CH_4$ volumes in the Tedlar bag and column headspace of Systems 1, 3, and 4.**

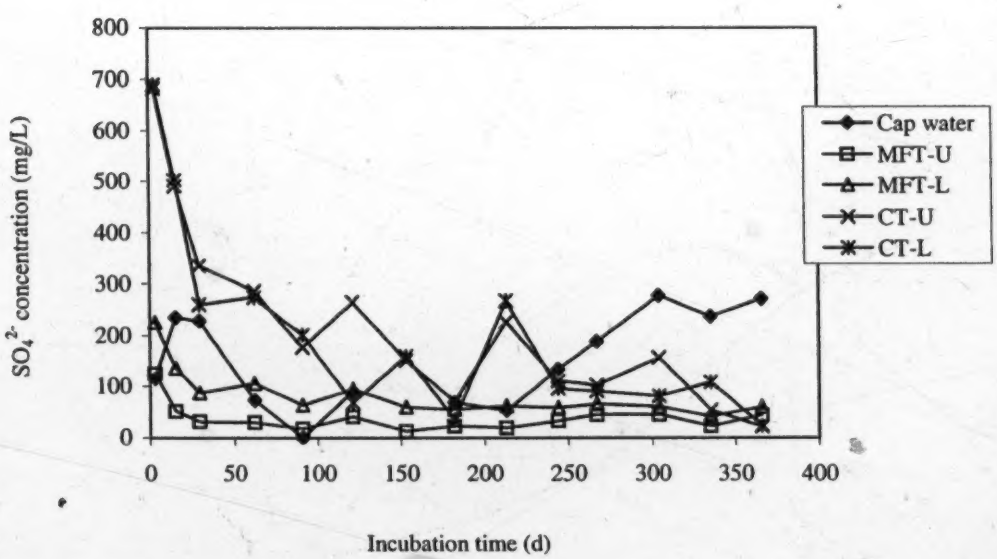



**Figure C-4. $SO_4^{2-}$ concentrations in System 1.**

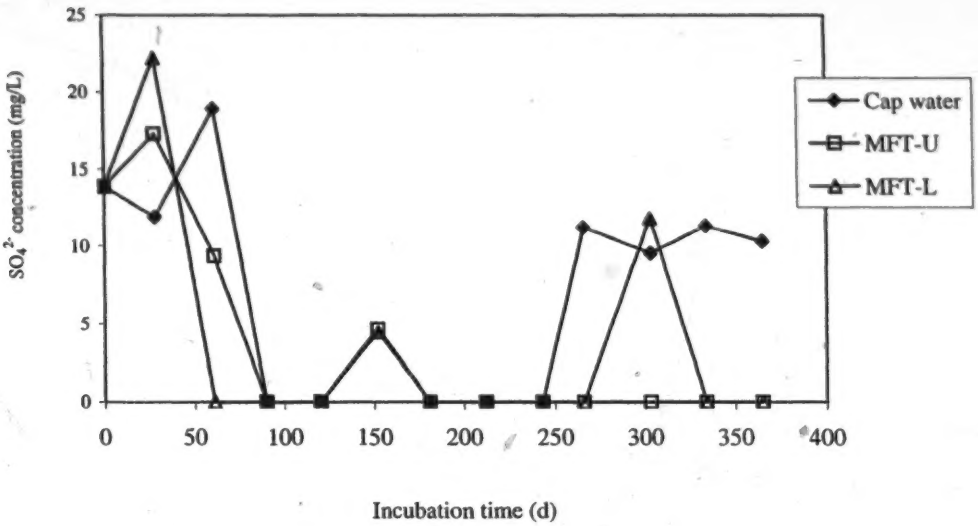



**Figure C-5. $SO_4^{2-}$ concentrations in System 3.**

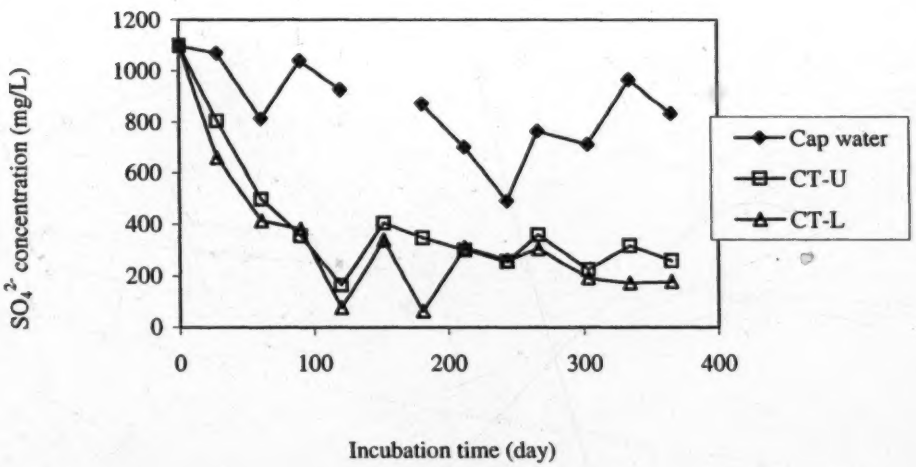



**Figure C-6. $SO_4^{2-}$ concentrations in System 4.**

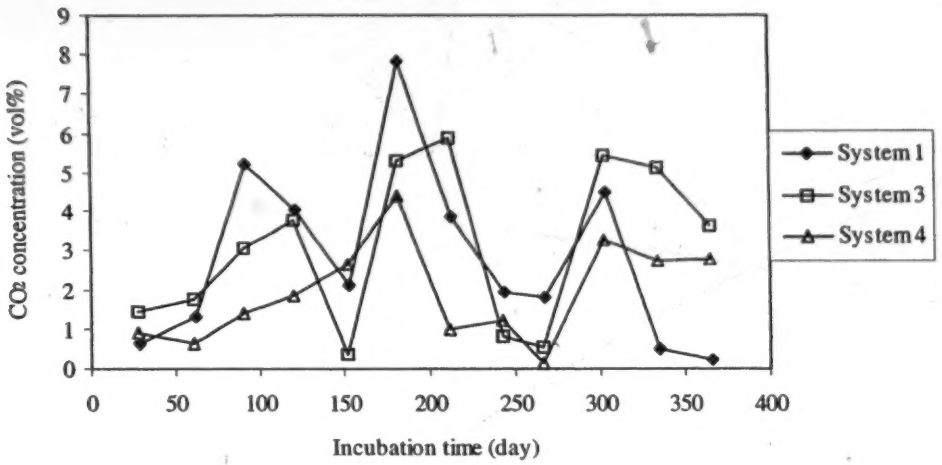



**Figure C-7. $CO_2$ concentrations in the headspace of Systems 1, 3, and 4.**

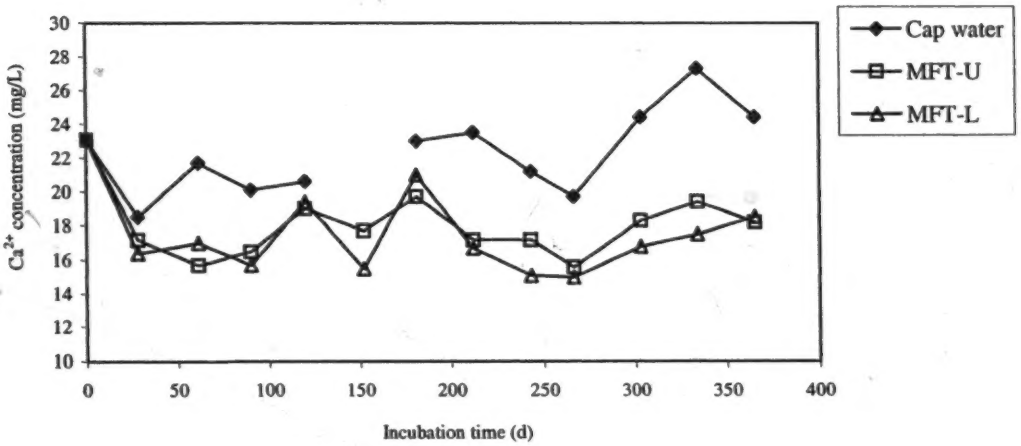



**Figure C-8. $Ca^{2+}$ concentrations in System 3.**

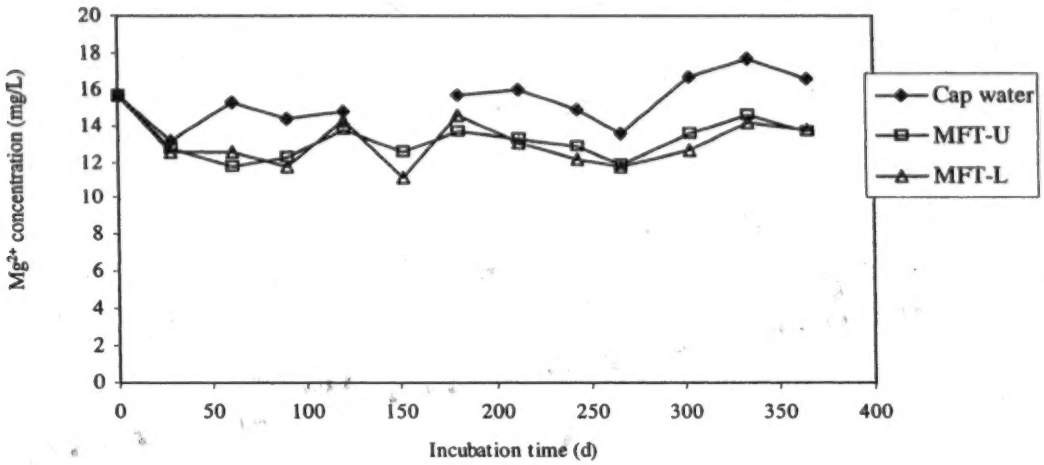



**Figure C-9. $Mg^{2+}$ concentrations in System 3.**

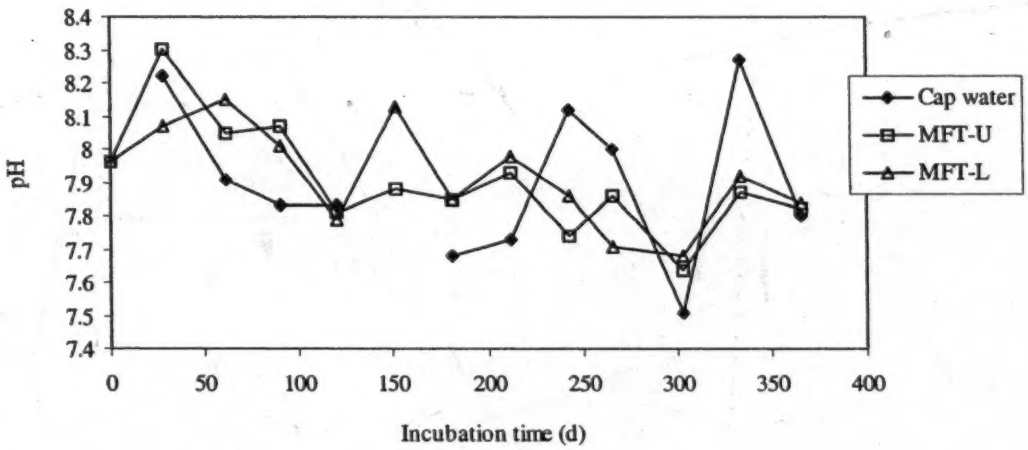



**Figure C-10. pHs in System 3.**

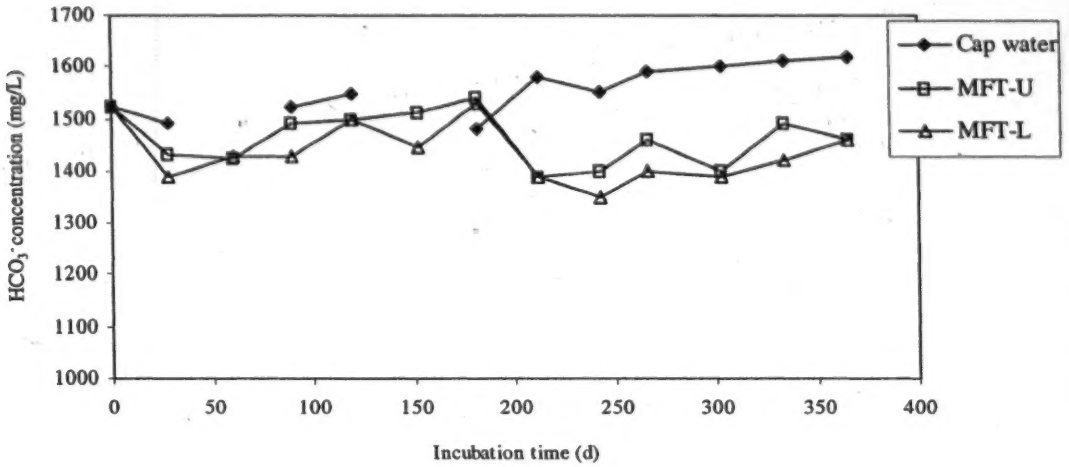



**Figure C-11. $HCO_3^-$ concentrations in System 3.**

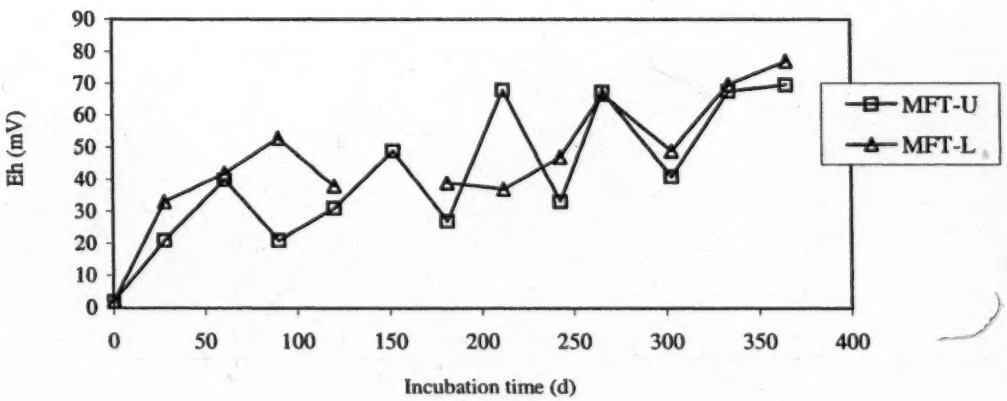



**Figure C-12. Redox potentials in System 3.**

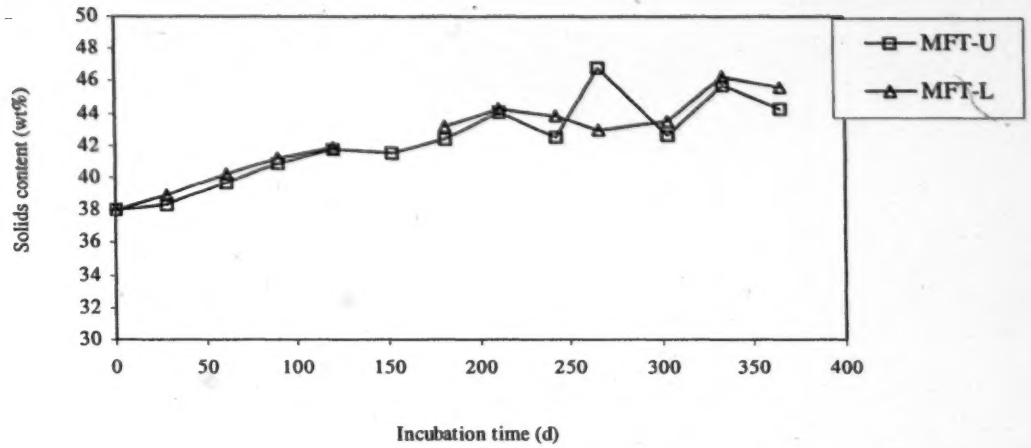



**Figure C-13. Solids contents in System 3.**

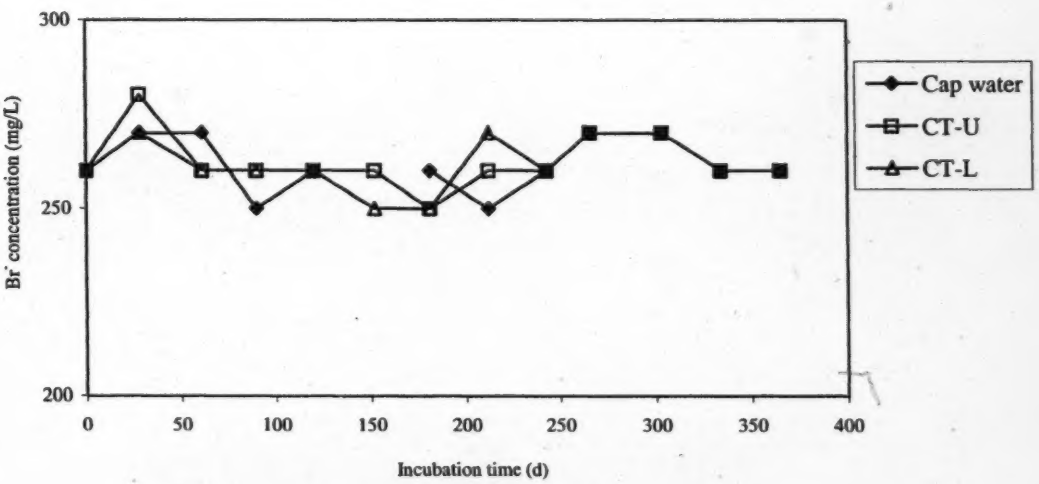



**Figure C-14. $Br^-$ concentrations in System 4.**

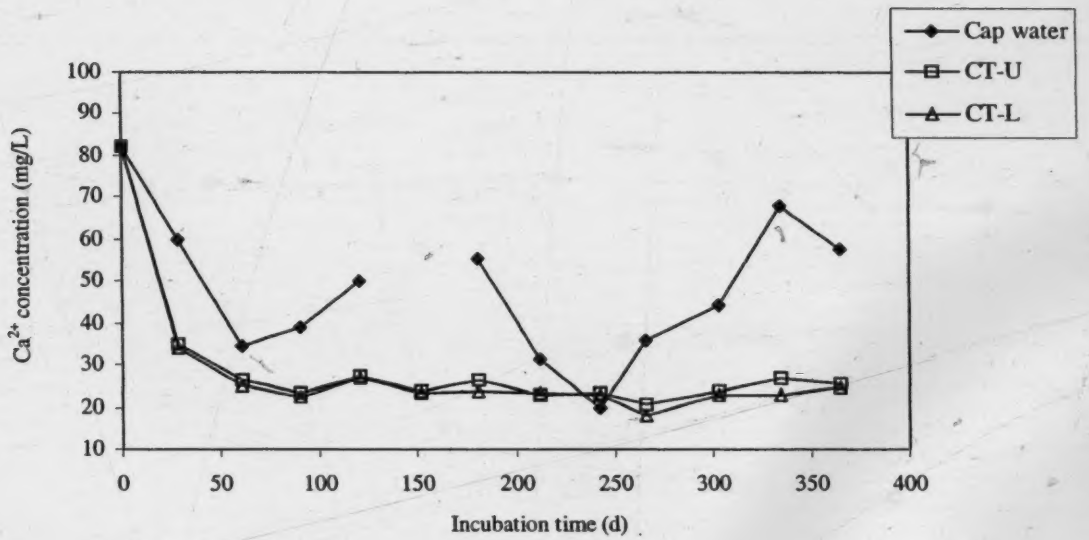



**Figure C-15. $Ca^{2+}$ concentrations in System 4.**

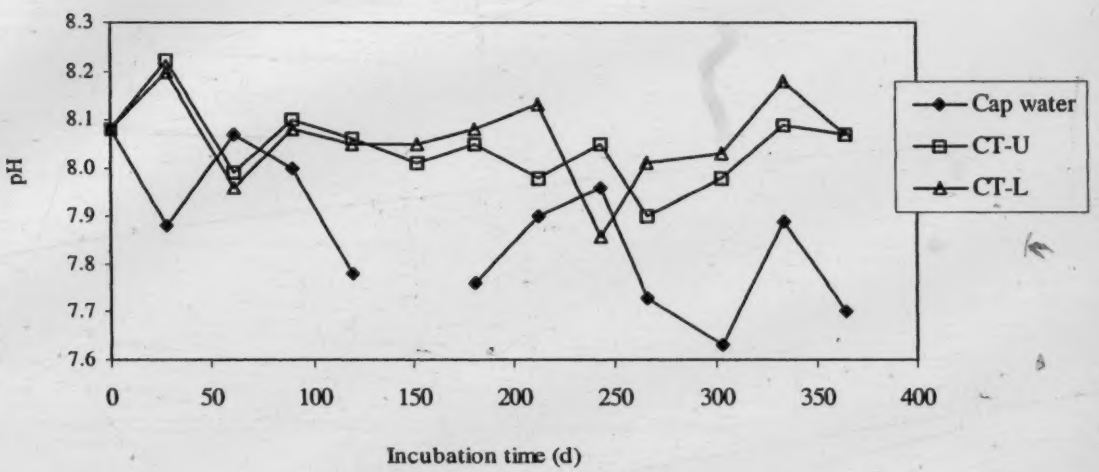



**Figure C-16. pHs in System 4.**

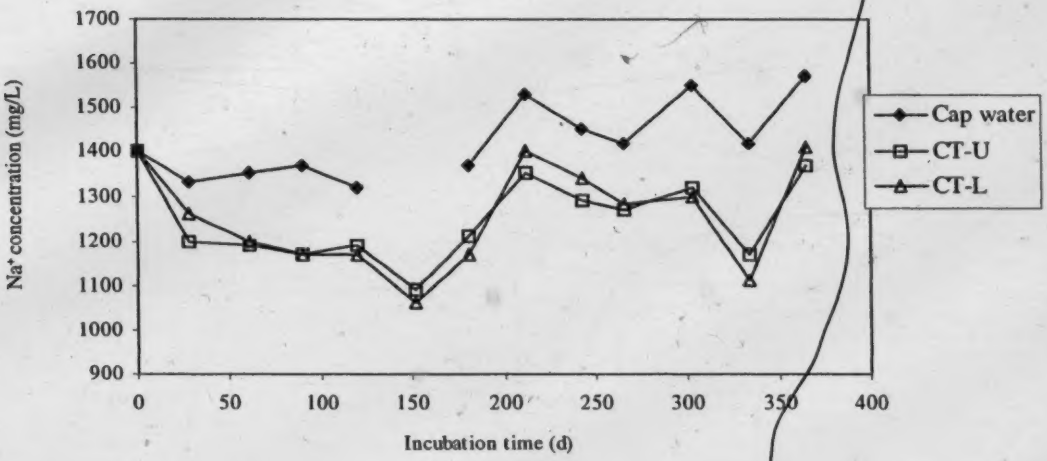



**Figure C-17. $Na^+$ concentrations in System 4.**

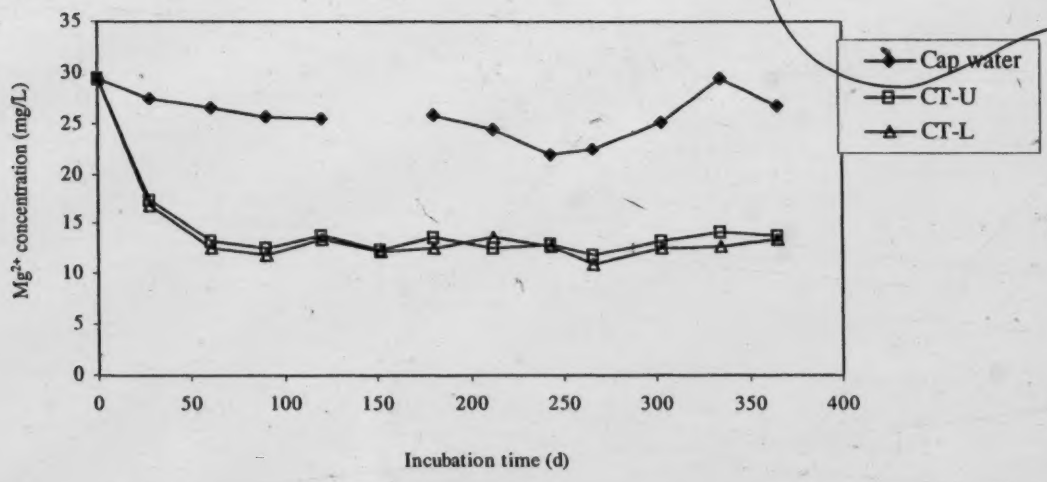



**Figure C-18. $Mg^{2+}$ concentrations in System 4.**

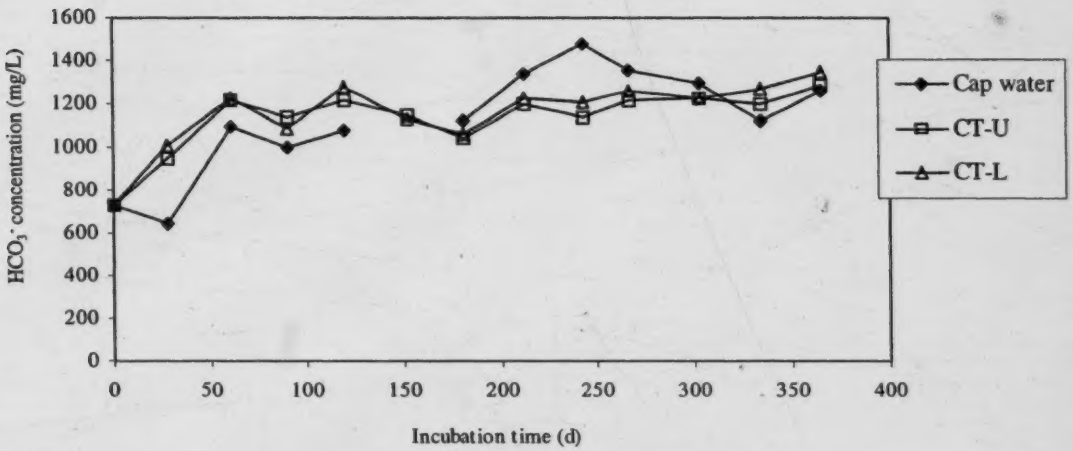



**Figure C-19. $HCO_3^-$ concentrations in System 4.**

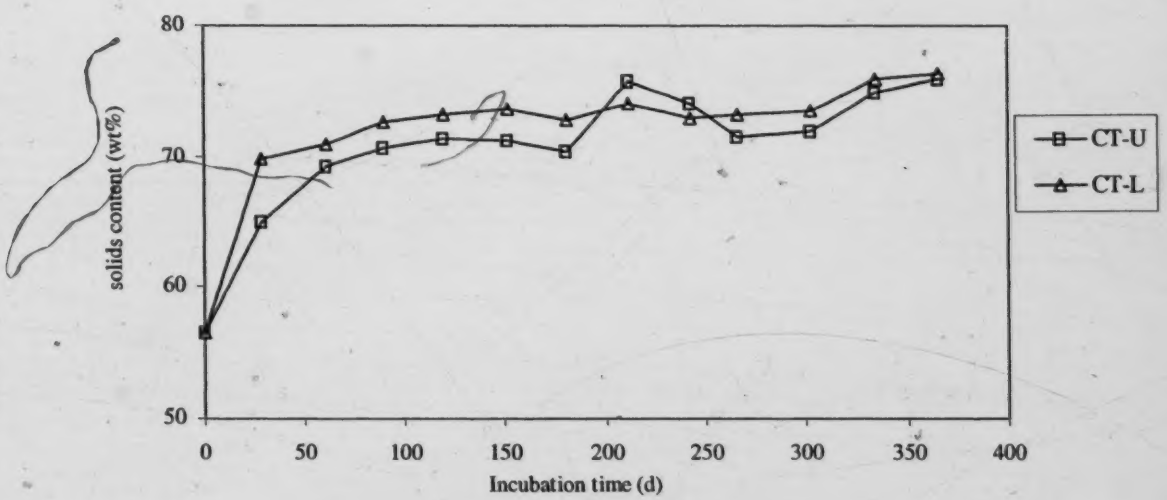



**Figure C-20. Solids contents in System 4.**

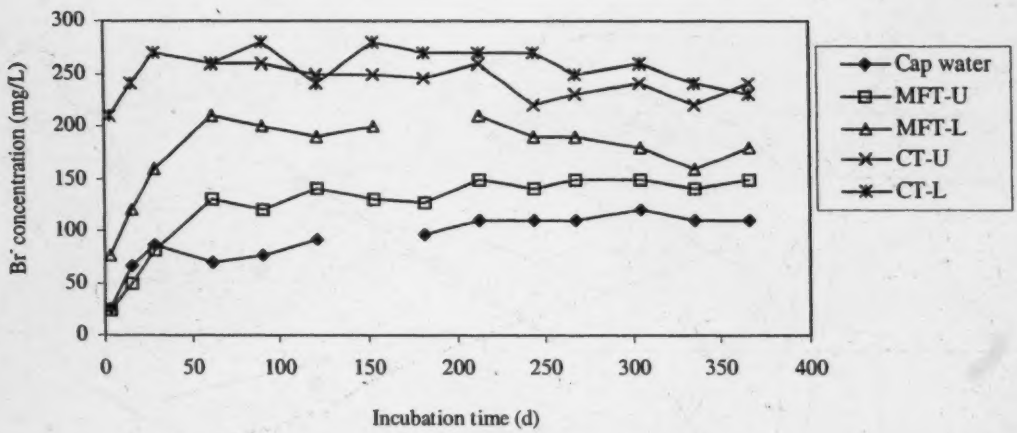



**Figure C-21. $Br^-$ concentrations in System 1.**

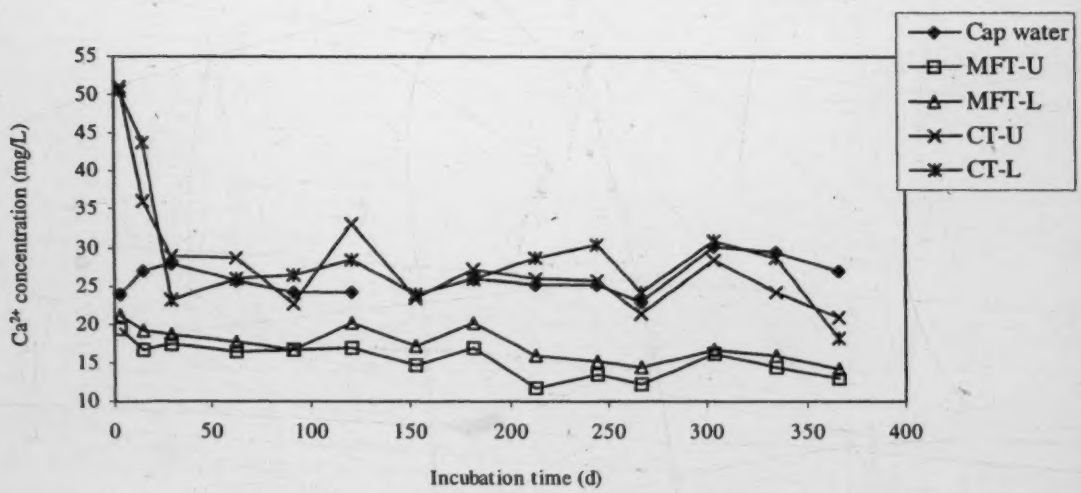



**Figure C-22. $Ca^{2+}$ concentrations in System 1.**

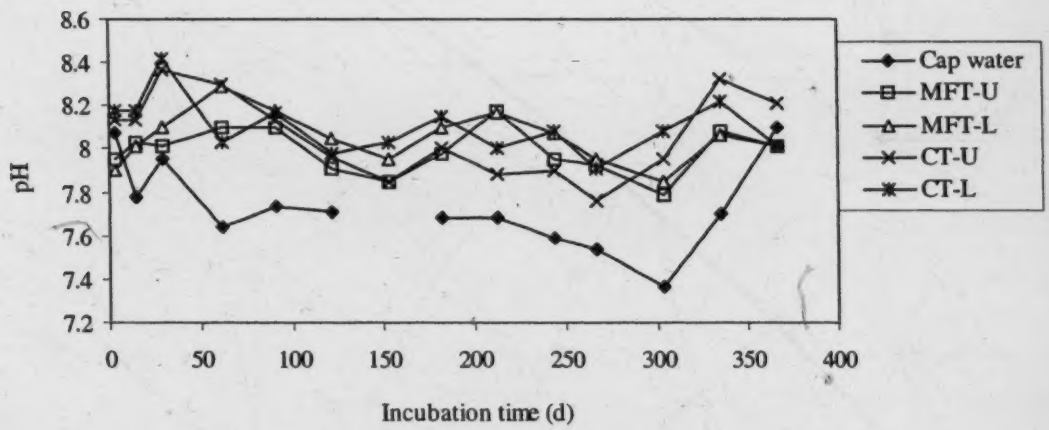



**Figure C-23. pHs in System 1.**

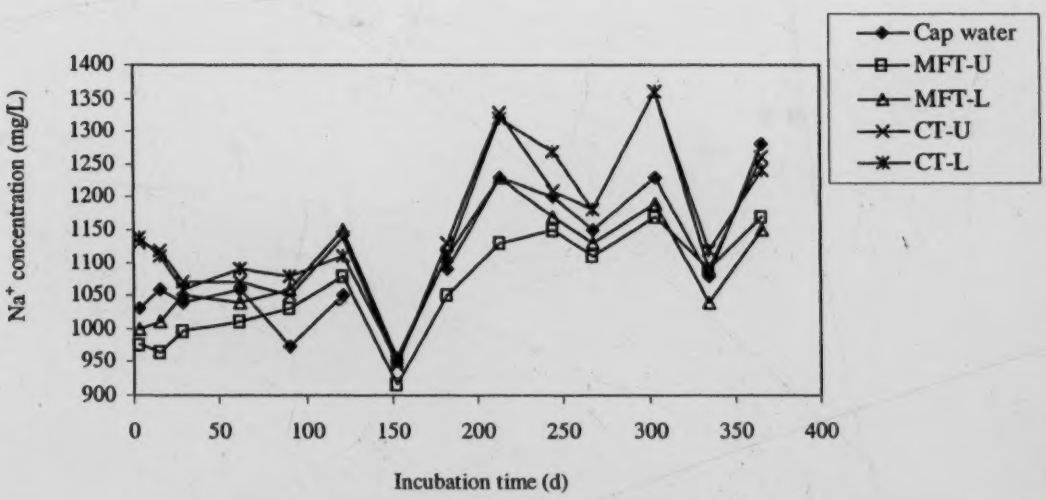



**Figure C-24. $Na^+$ concentrations in System 1.**

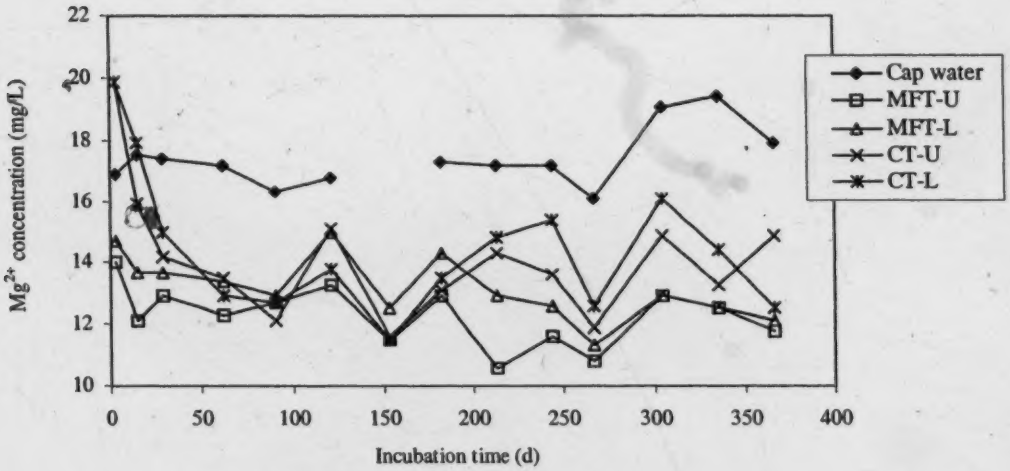



**Figure C-25. $Mg^{2+}$ concentrations in System 1.**

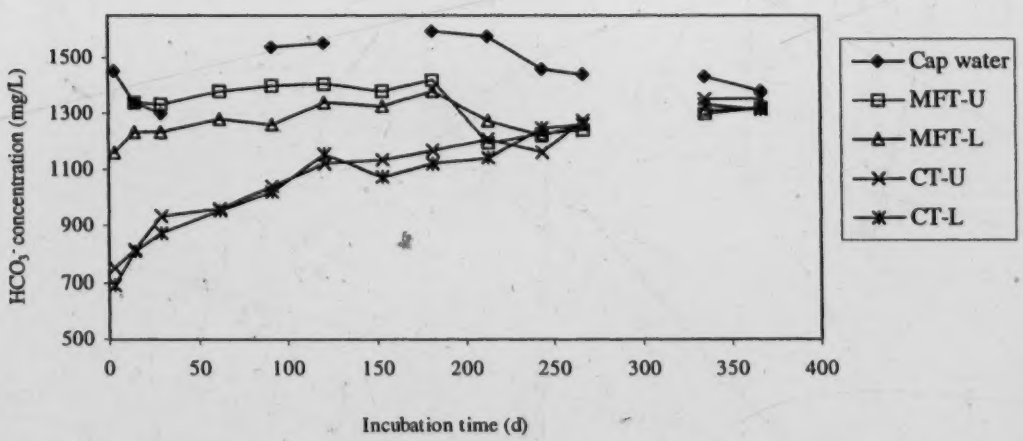



**Figure C-26. $HCO_3^-$ concentrations in System 1.**

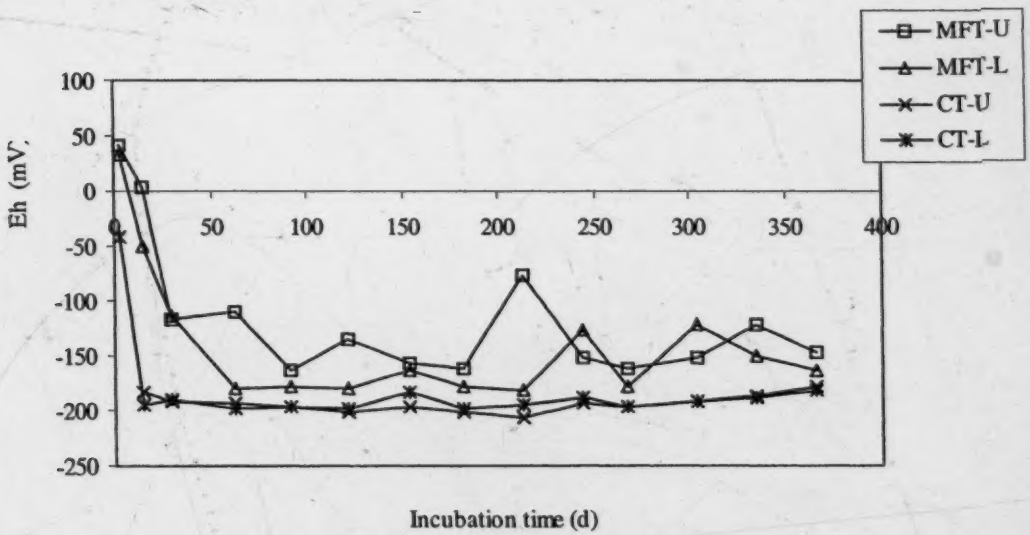



**Figure C-27. Redox potentials in System.**

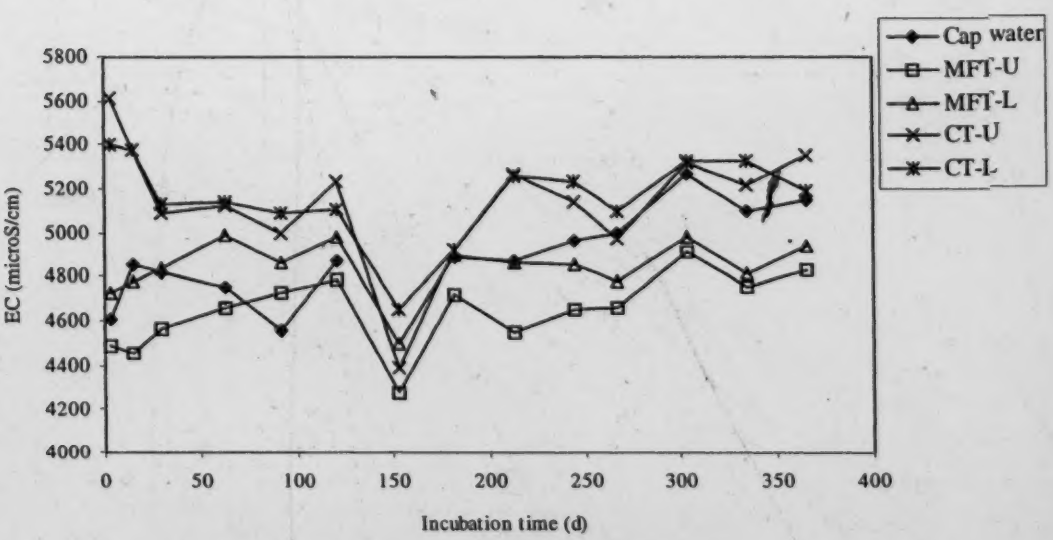



**Figure C-28. Electrical conductivities in system 1.**

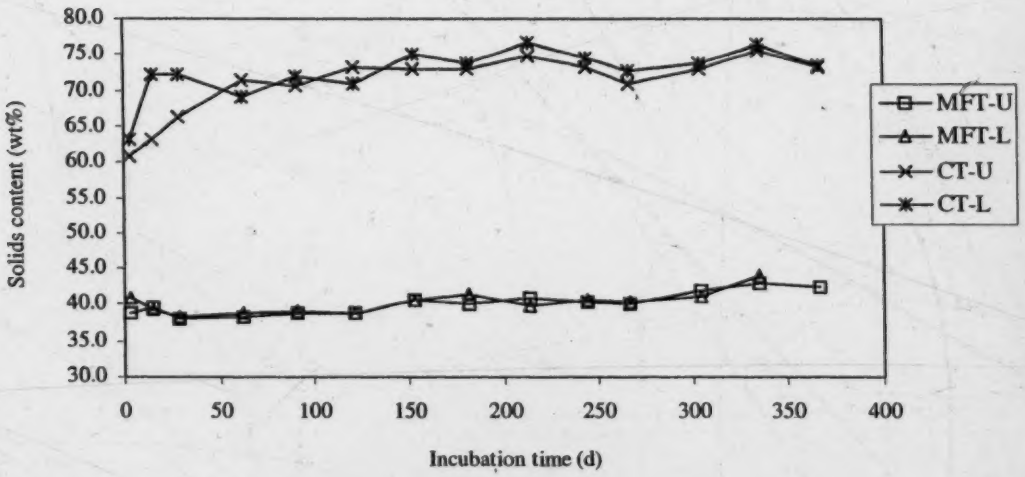



**Figure C-29. Solids contents in System 1.**

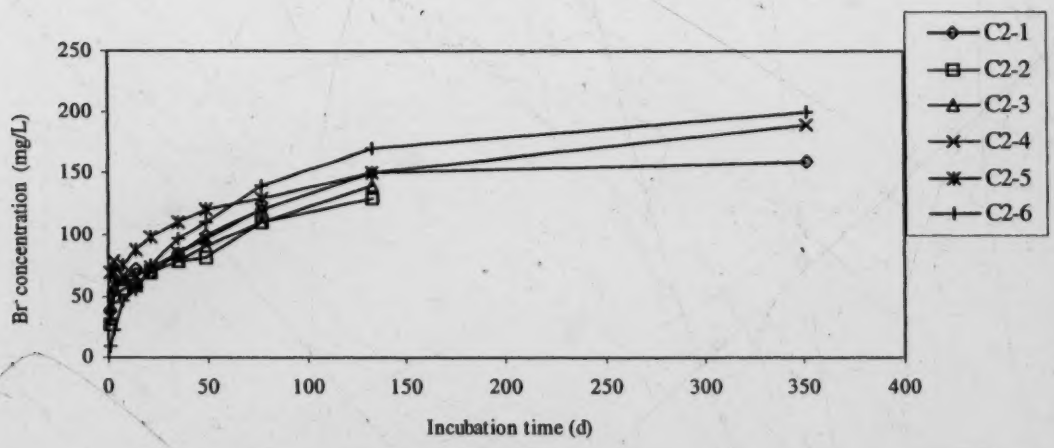



**Figure C-30. $Br^-$ concentrations in the cap water of System 2.**

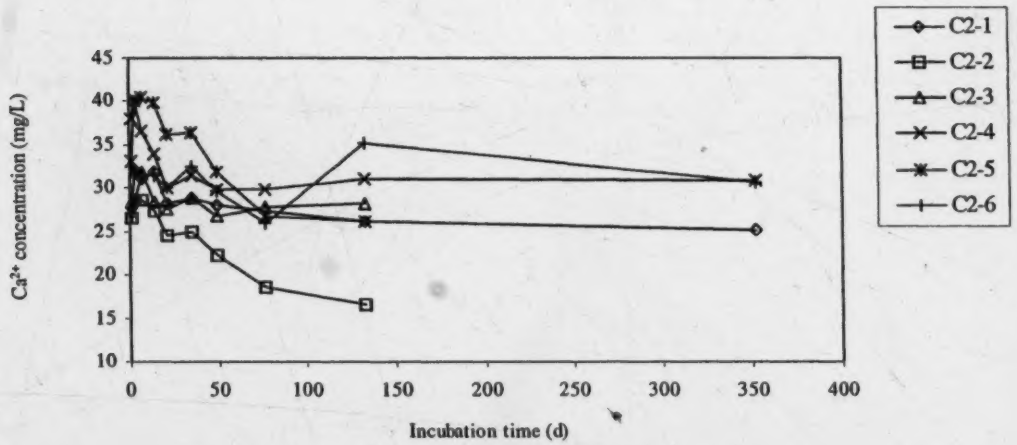



**Figure C-31. $Ca^{2+}$ concentrations in the cap water of System 2.**

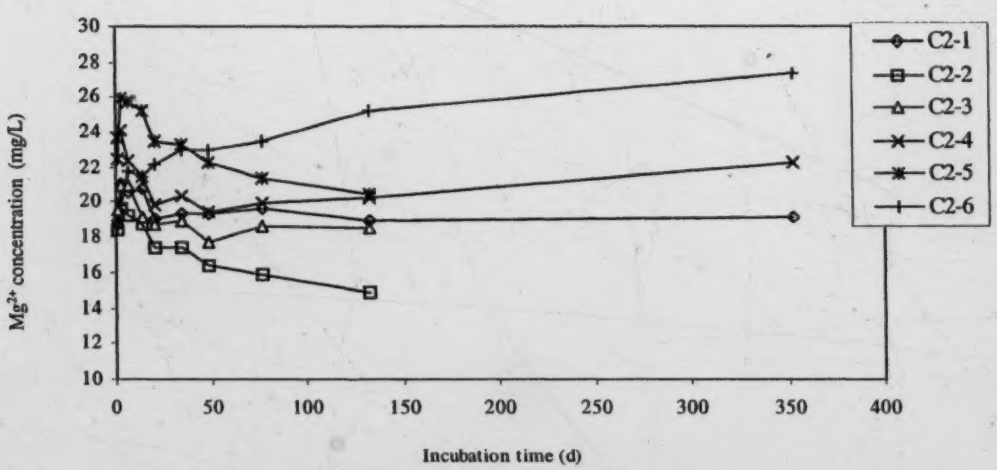



**Figure C-32. $Mg^{2+}$ concentrations in the cap water of System 2.**

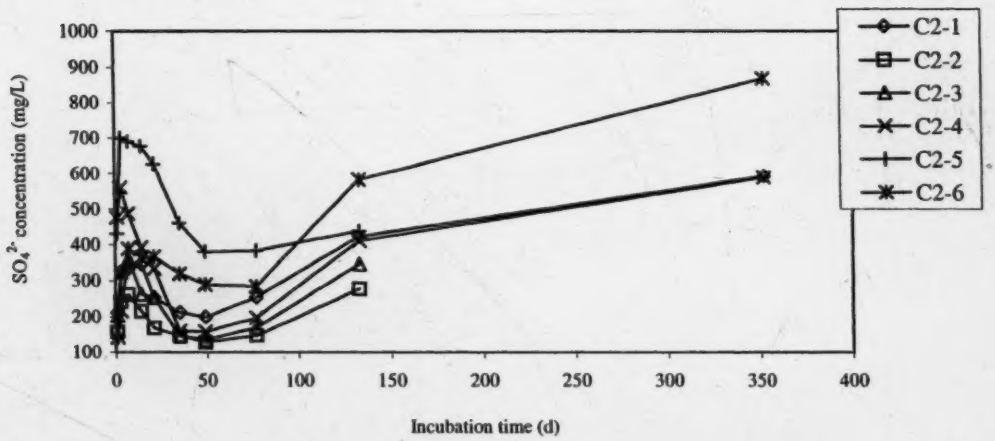



**Figure C-33. $SO_4^{2-}$ concentrations in the cap water of System 2.**

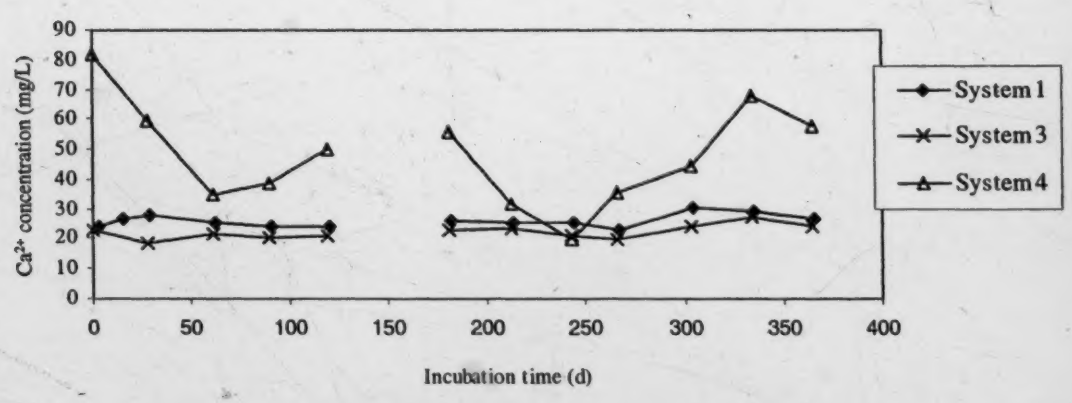



**Figure C-34. $Ca^{2+}$ concentrations in the cap water of Systems 1, 3, and 4.**

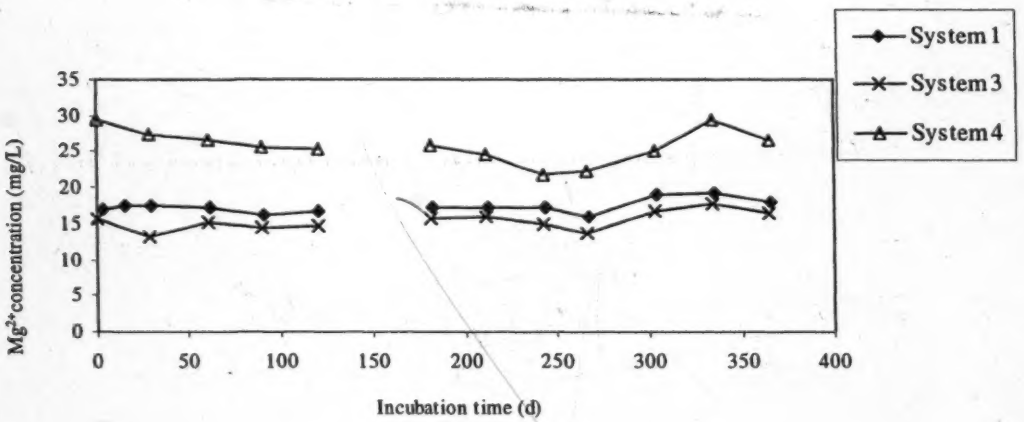



**Figure C-35. $Mg^{2+}$ concentrations in the cap water of Systems 1, 3, and 4.**

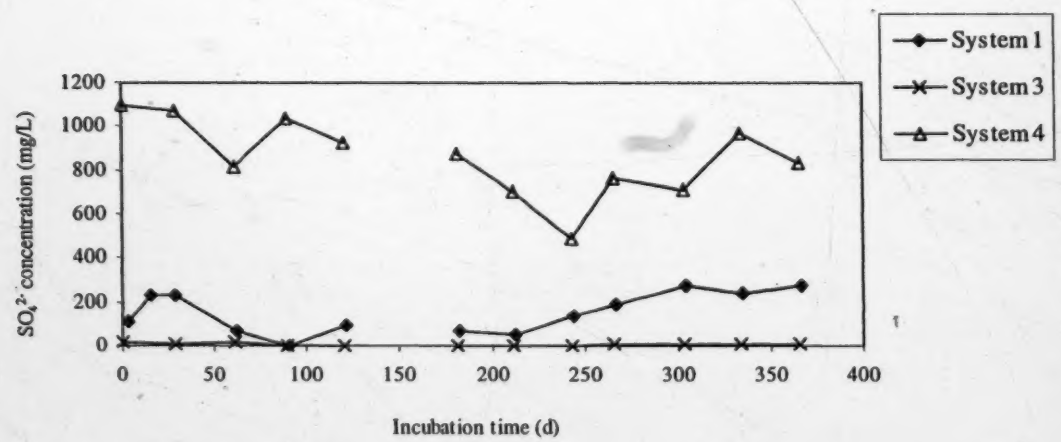



**Figure C-36. $SO_4^{2-}$ concentrations in the cap water of Systems 1, 3, and 4.**

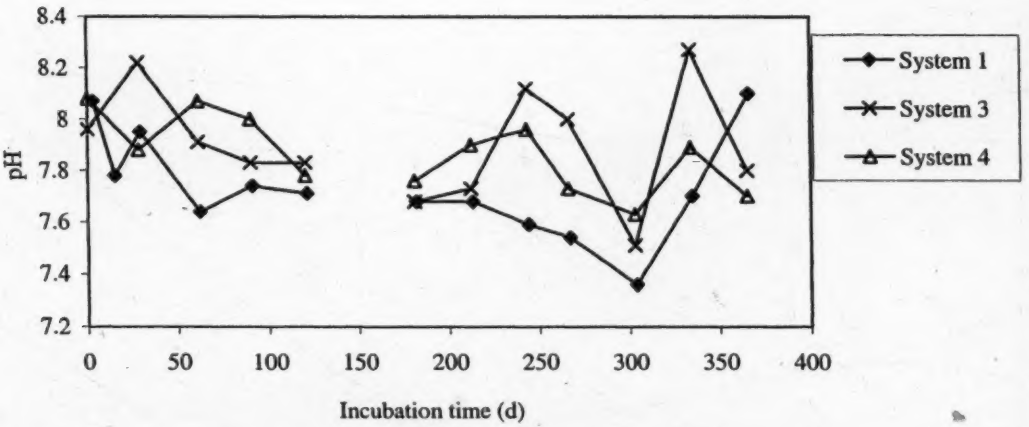



**Figure C-37. pHs in the cap water of Systems 1, 3, and 4.**

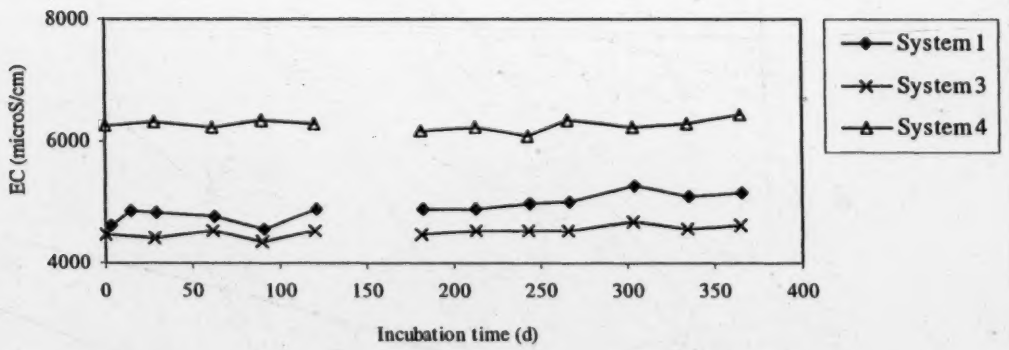



**Figure C-38. Electrical conductivities in the cap water of Systems 1, 3, and 4.**

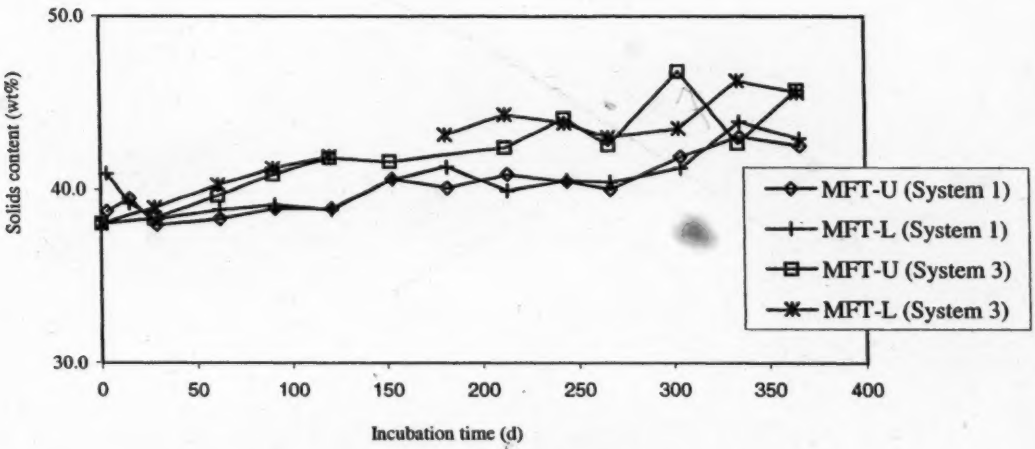



**Figure C-39. Solids contents of MFT in Systems 1 and 3.**

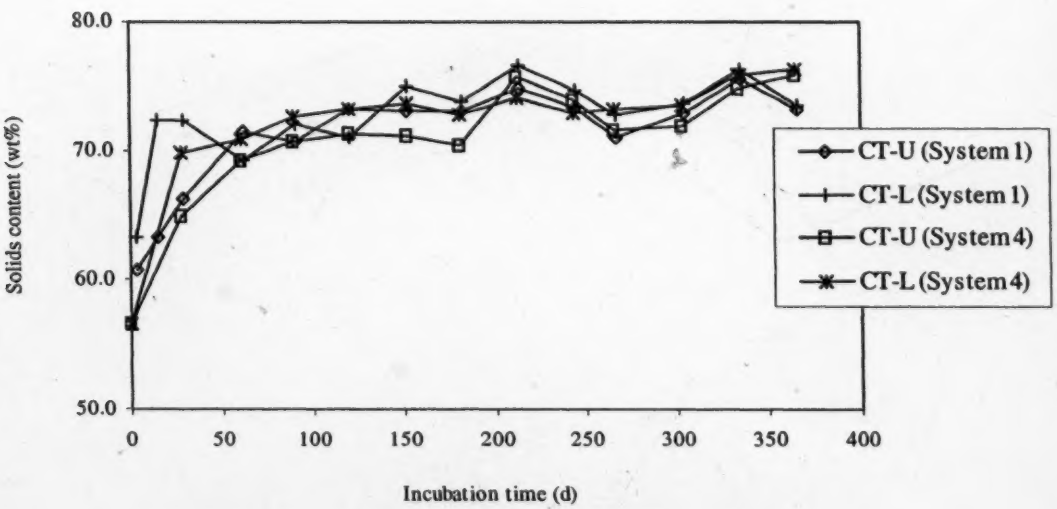



**Figure C-40. Solids contents of CT in Systems 1 and 4.**

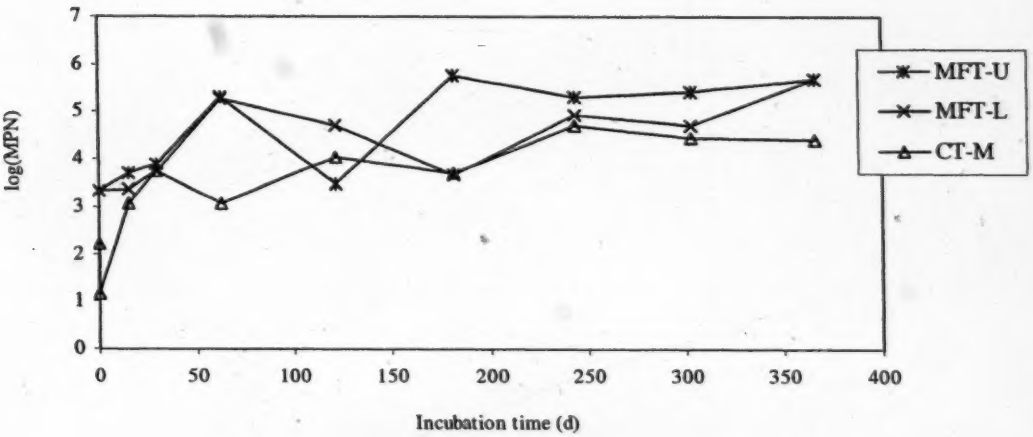



**Figure C-41. MPN of Methanogens in System 1.**

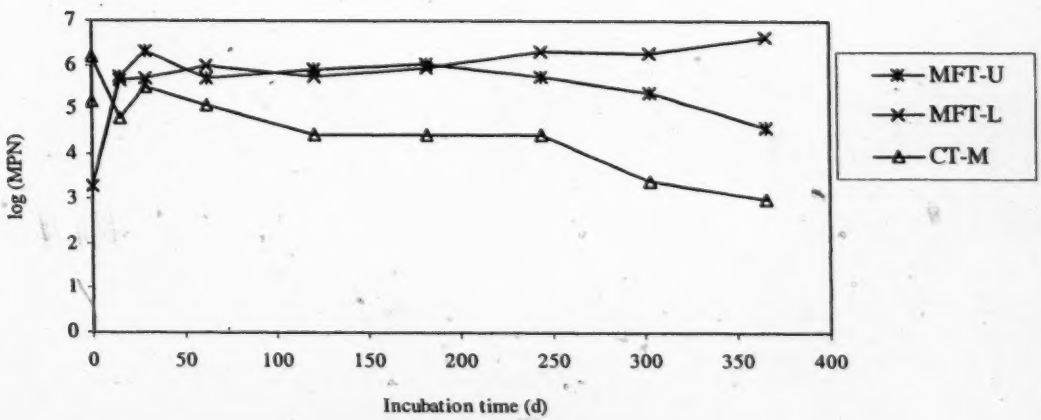



**Figure C-42. MPN of SRB in System 1.**

**Table C-1. Comparison of $CO_2$ concentrations in the headspace of System 1 between the calculated concentrations by Henry's law and the measured concentrations by GC.**

| Column | $t_{incu}$ (d) | $[H_2CO_3^*]$ in cap water (M) | Calculated $Pco_2$ in headspace (atm) | Calculated $CO_2$ concentration in headspace (vol%) | Measured $CO_2$ concentration in headspace (vol%) |
|---|---|---|---|---|---|
| C1-4 | 29 | 5.6E-04 | 1.8E-02 | 1.76 | 0.64 |
| C1-5 | 62 | NA | NA | NA | 1.29 |
| C1-6 | 91 | 1.1E-03 | 3.4E-02 | 3.38 | 5.23 |
| C1-12 | 121 | 1.2E-03 | 3.6E-02 | 3.64 | 4.03 |
| C1-8 | 153 | NA | NA | NA | 2.10 |
| C1-9 | 182 | 1.3E-03 | 4.0E-02 | 4.02 | 7.82 |
| C1-10 | 213 | 1.3E-03 | 4.0E-02 | 3.98 | 3.88 |
| C1-11 | 244 | 1.4E-03 | 4.5E-02 | 4.52 | 1.96 |
| C1-13 | 267 | 1.6E-03 | 5.0E-02 | 5.01 | 1.78 |
| C1-14 | 304 | NA | NA | NA | 4.50 |
| C1-15 | 335 | 1.1E-03 | 3.4E-02 | 3.44 | 0.50 |
| C1-16 | 366 | 4.2E-04 | 1.3E-02 | 1.32 | 0.24 |

1). Assuming the total gas pressure in the headspace is 1 atm.
2). NA=not available.

**Table C-2. Empirical estimations of masses of solids and porewater per unit volume of MFT or CT at specific solids content.**

| Solids content of MFT or CT (wt%) | ρ (density of MFT or CT) (g/L) | Mass of solids (g/L of MFT or CT) | Volume of porewater (mL/L of MFT or CT) | Solids content of MFT or CT (wt%) | ρ (density of MFT or CT) (g/L) | Mass of solids (g/L of MFT or CT) | Volume of porewater (mL/L of MFT or CT) |
|---|---|---|---|---|---|---|---|
| 38 | 1308 | 497 | 811 | 58 | 1562 | 906 | 656 |
| 39 | 1319 | 514 | 805 | 59 | 1577 | 930 | 647 |
| 40 | 1330 | 532 | 798 | 60 | 1592 | 955 | 637 |
| 41 | 1341 | 550 | 791 | 61 | 1608 | 981 | 627 |
| 42 | 1352 | 568 | 784 | 62 | 1624 | 1007 | 617 |
| 43 | 1364 | 586 | 777 | 63 | 1641 | 1034 | 607 |
| 44 | 1375 | 605 | 770 | 64 | 1658 | 1061 | 597 |
| 45 | 1387 | 624 | 763 | 65 | 1675 | 1089 | 586 |
| 46 | 1399 | 644 | 756 | 66 | 1693 | 1117 | 576 |
| 47 | 1411 | 663 | 748 | 67 | 1711 | 1146 | 565 |
| 48 | 1424 | 683 | 740 | 68 | 1729 | 1176 | 553 |
| 49 | 1436 | 704 | 733 | 69 | 1748 | 1206 | 542 |
| 50 | 1449 | 725 | 725 | 70 | 1767 | 1237 | 530 |
| 51 | 1462 | 746 | 717 | 71 | 1786 | 1268 | 518 |
| 52 | 1476 | 767 | 708 | 72 | 1806 | 1301 | 506 |
| 53 | 1489 | 789 | 700 | 73 | 1827 | 1334 | 493 |
| 54 | 1503 | 812 | 692 | 74 | 1848 | 1367 | 480 |
| 55 | 1517 | 835 | 683 | 75 | 1869 | 1402 | 467 |
| 56 | 1532 | 858 | 674 | 76 | 1891 | 1437 | 454 |
| 57 | 1547 | 882 | 665 | 77 | 1914 | 1473 | 440 |

1). Assuming:

a). The density of water is 1 g/mL.

b). The mass of MFT or CT is the sum of the masses of solids and porewater.

c). $\rho = (1/(1-0.0062 \times wt\%)) \times 1000$ (g/L of MFT or CT).

2). Sample calculation (at solids content of 38 wt%):

a). The mass of solids = $\rho \times wt\% = 1308 \times 38\% = 497$ (g/L of MFT or CT).

b). The mass of porewater = 1308-497=811 (g/L of MFT or CT).

c). The volume of porewater =811 (mL/L of MFT or CT).

**Table C-3. Empirical estimation of released and still remaining MFT waters, in originally 1 L MFT with initial MFT solids content of 38 wt%.**

| MFT | | | Waters | | |
|---|---|---|---|---|---|
| Solids content (wt%) | ρ (density) (g/mL) | Volume (mL) | MFT porewater (mL) | Released MFT water (mL) | Remaining MFT porewater (vol%) |
| 38 | 1.308 | 1000 | 811 | 0 | 100 |
| 39 | 1.319 | 967 | 778 | 34 | 96 |
| 40 | 1.330 | 935 | 746 | 65 | 92 |
| 41 | 1.341 | 904 | 715 | 96 | 88 |
| 42 | 1.352 | 876 | 687 | 125 | 85 |
| 43 | 1.364 | 848 | 659 | 152 | 81 |
| 44 | 1.375 | 822 | 633 | 178 | 78 |
| 45 | 1.387 | 797 | 608 | 204 | 75 |
| 46 | 1.399 | 773 | 584 | 228 | 72 |
| 47 | 1.411 | 750 | 561 | 251 | 69 |
| 48 | 1.424 | 728 | 539 | 273 | 66 |
| 49 | 1.436 | 706 | 517 | 294 | 64 |
| 50 | 1.449 | 686 | 497 | 314 | 61 |

1). Percent of remaining porewater (vol%) is relative to the total MFT porewater volume at solids content of 38wt%,

2). Table C-3 is adapted from the relevant table, which is provided by Dr. MacKinnon in Syncrude.